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Power and Production

OF the various factors influencing the economic well-being of Britain to-day, many are not entirely within the control of the people of this country. All the more reason, therefore, why we should make strenuous efforts to arrive at a solution of those problems which we can tackle. There is little doubt that an increase in coal production and an increase in industrial productivity generally are two essential requirements if we are to make any advance in our struggle against present day conditions. It may be argued that the first requirement named is merely a special case of the second, but it has been singled out because it is probably the most important single factor, in that everything else is related to it.

Before the war we were able to export considerable quantities of coal which were surplus to our requirements, but to-day the boot is on the other foot and we are now among the importers. Many figures have been quoted on the relative positions to-day and before the war but they only serve to disguise the simple fact that supply is not equal to demand. The convenience of the electric fire has had much to do with the increase in its use in the home, but in the minds of many people it is a poor substitute for a coal fire, and a little extra coal in the domestic coal-place would result in a lessening of the demand for electric power. And that is surely no mean factor in these days when staggered working hours and other schemes are being discussed as a means of avoiding complete shutdowns in industry because of the shortage of electrical generating plant. It is little use industry adopting increased mechanisation, as advocated by every productivity team which has visited the U.S.A., if the resulting power demand is going to dislocate the whole electrical generating and distribution system. Better than any scheme for reducing unwanted electricity demand by banning the sale of electric fires, water heaters, etc., is, surely, the provision of something which will reduce the use made of those in existence—and that something is coal. The output per miner is said to be higher to-day than it was before the war, but that factor is offset by the fall in man-power since pre-war days. Increasing mechanisation may increase productivity per man, but if it is overshadowed by the reduction in the number of workers there can be no progress. Although the working conditions and rewards are a great improvement on those of former years, the industry is not attracting a sufficient number of younger workers to make good the wastage due to retirement. What then can one make of the attitude of workers in the industry to the introduction of foreign labour? If Britons will not work in British mines, why stop people who are willing to do so? It appears that the miners have no more faith in the N.C.B. than they had in the old private owners.

A further effect of the need to import coal—an indirect one, but, nevertheless, a very important one—concerns imports of iron ore. Formerly, when we used to export coal, the ships which carried the coal brought back iron ore with which to feed our blast furnaces. To-day, with our shipping capacity stretched to the limit by such factors as the Korean War, this use of ships for carrying coal *to* Britain instead of *from* Britain, has had an adverse effect on the iron ore position and, in consequence, on the production of iron and steel—a matter of vital importance.

Turning now to the question of general productivity, we make no apology for having, in these columns, dwelt on the importance of the human factor, because we consider it is undoubtedly the most important one. The latest Productivity Report,* and, incidentally, the longest to date, deals with the American brassfoundry industry, and again stresses the greater incentive to earn in the United States. It is emphasized that the American brassfoundry industry is not of the up-to-date highly-mechanised type so many people associate with the United States. Actually it is similar to that in Great Britain, being made up of a large number of relatively small foundries. On the technical side, the team considers that Britain is ahead of American standards, but when it comes to production, the output per man-hour in the U.S. is higher than in British plants of similar type and size. As stated earlier, the Team considers that the chief factor is the greater incentive to earn in America, where a high level of both income and production has resulted in a standard of living which far exceeds anything so far known. For example, an American worker can earn a pair of shoes in about 7 hours and a suit in three days, as compared with 15 hours and three weeks, respectively, in this country. It is felt that in this country we value security and leisure more highly than a better standard of living.

A second reason for the higher American productivity is given as the greater use of mechanisation, in particular to the extensive use of simple roller conveyors and other means of servicing the operatives. The inexpensive overhead mechanical handling systems of the mono-rail type have been referred to in a number of earlier reports, and attention is drawn here to the fact that their success depends to a large extent on the development of a flat-flange I-beam with raised tread, made from high carbon steel with a minimum Brinell hardness of 260, which has eliminated many of the difficulties encountered with bearings in carriers running on bevelled-flange beams.

Melting is generally carried out in a separate shop, using gas or oil-fired lift-out crucible furnaces. Silicon carbide crucibles seem to be preferred and give some 50–70 heats when melting 85/5/5/5 gunmetal. Crucibles of molten metal are always withdrawn from the furnace by an overhead electric mono-rail crane, using shanks

* "The Brassfoundry." Copies may be obtained from the Anglo-American Council on Productivity, 21, Tothill St., London, S.W.1., or from either the Association of Bronze and Brass Founders (Secretaries Heathcote and Coleman), 25, Bennett's Hill, Birmingham, 2, or the Secretary, The National Brassfoundry Association, 30, Waterloo St., Birmingham, 2. Price: 7s. 6d. post free.

which appear to be less likely to strain the crucible than the ring and wedge type commonly used in Britain.

Great importance is attached to accurate pattern equipment, and it is common practice to standardise on match-plates, complete with all ingates, runners and dirt traps even for such short runs as 50 off. Aluminium alloy match-plates are preferred, whilst plaster, wood or synthetic resin plates are used for short runs. As in the case of the ironfoundry industry, pattern requirements are economically met by outside firms specialising in this work.

Moulding boxes with round and elongated pin bushes are widely used in America and assist productivity by facilitating the rapid opening and closing of moulds. Very few foundries were seen using dry-sand moulds; large castings were generally made in green sand and then skin-dried, or alternatively, the mould was built up from oil-sand sections.

Other highlights noted include a 20-in. unbreakable high-speed cutting-off wheel, the use of gypsum risers sleeves to promote better feeding, and the "C" process of moulding which is attracting a good deal of interest. This process was developed in Germany during the war and half-mould shells are made by allowing a sand/phenolic resin mixture to fall on to a hot metal pattern. For pouring, the half-mould shells are backed-up by steel shot.

A further factor of some importance, and one in which the co-operation of the customer will be necessary, is the reduction in the number of alloys cast. These seem to have been reduced to about six in the U.S. and of the castings seen by the Team in the foundries visited, 75% were made from 85/5/5/5 gunmetal. This undoubtedly contributes to the higher American productivity and provides a promising field of endeavour in our efforts to increase productivity on this side of the Atlantic.

New and Revised British Standards

Bronze Welding by Gas. (B.S.1724 : 1951)

This is a supplementary standard to B.S.1723 'Brazing' and has been prepared as part of the programme of welding standards authorised by the Welding Industry Standards Committee of the Institution. In addition a request was received from the Codes of Practice Committee convened by the Institution of Mechanical Engineers and the Institution of Heating and Ventilating Engineers for standards for welded joints in galvanized pipes and copper pipes. Accordingly, the standard gives not only the requirements applicable to the process generally but also individual requirements in respect of various applications.

Under general requirements are included a description of the parent metals to which the process can be applied, specifications for filler metals and a system of testing procedure. The application of the process to specific parent metals, namely, copper, mild steel, galvanized mild steel, cast iron, malleable iron and dissimilar metals is dealt with in individual sections, particular attention being given to the details of the joints. Included as an appendix is a useful set of photographs illustrating neutral, oxidizing and carburizing oxy-acetylene welding flames.

Steel Tubes for Mechanical, Structural, and General Engineering Purposes. (B.S.1775 : 1951)

This new standard is the third of a series for steel tubes for general engineering purposes, the other two being B.S.980, 'Steel tubes for automobile purposes,' and B.S.1717, 'Steel tubes for cycle and motor cycle purposes.'

The standard covers plain carbon steel tubes not exceeding 16 in. outside diameter, of the following types: Hot finished welded (referred to as type HFW); hot finished seamless (referred to as type HFS); cold drawn seamless (referred to as type CDS); electric resistance butt-welded (referred to as type ERW); cold drawn electric resistance butt-welded (referred to as type CEW); oxy-acetylene butt-welded (referred to as type OAW). Tubes of types HFW, HFS, CDS and OAW are specified in four grades, viz. 11, 13, 15 and 20, the numbers indicating the minimum yield strength. Tubes of type ERW are similarly specified in Grades 11, 15 and 20, while those of type CEW are specified in Grades 11, 15, 24 and 28.

Part One includes a number of general clauses applicable to all types of tubes, dealing with such matters as material, straightness, lengths, galvanizing, methods of test and inspection. Each type of tube is then dealt with in detail in a separate section which includes such items as tolerances and mechanical properties. At the end of the standard are tables of standard sizes of hot finished welded and hot finished seamless tubes. A further table gives the properties of the tabulated sizes.

Carbon Steel Castings for Surface Hardening. (B.S.1760 : 1951)

This standard, one of a series of standards being prepared for steel castings for general engineering purposes, covers the requirements for carbon steel castings for surface hardening by a local heating and quenching process. Two grades are specified:—

Grade A is required to show a tensile strength of 40 ton/sq. in. and a surface hardness after hardening of 550 DPN.

Grade B is required to show a tensile strength of 45 ton/sq. in. and a surface hardness after hardening of 600 DPN.

The full chemical composition is specified for both grades. Inspection and testing are covered in detail and an appendix gives recommendations on the repair of castings by metal-arc welding.

Copies of these standards may be obtained from the British Standards Institution, Sales Department, 24, Victoria Street, London, S.W.1., at the following prices: B.S.1724, 3s. 6d.; B.S.1760, 2s.; and B.S.1775, 3s.; all post free.

The Steel Company of Wales (Lysaght Works) Ltd.

The Board of the Steel Company of Wales, Ltd., has decided that as from October 1st, 1951, the Orb Works at Newport, which was formerly operated as the Steel Company of Wales (Lysaght Works), Ltd., is to be operated as the Lysaght Division of the Steel Company of Wales. No changes in management or policy are involved.

Metallurgical Considerations in the Use of Aluminium for Cable Sheathing—I

By A. Latin, Ph.D., M.Eng.

British Insulated Callender's Cables, Ltd.

A number of interesting problems have arisen in the development of aluminium sheathing for electric cables, and in this article the author discusses a number of metallurgical factors involved, dealing in this issue with general metallurgical considerations and the possible methods of sheathing.

THE use of aluminium in place of lead for the sheathing of electric cables has reached considerable proportions and this article deals with some of the more basic technical factors involved in this use in comparison with lead.

It is worth while firstly to consider, briefly, some of the factors concerned in any question of replacement of one material by another for any given purpose. Some at least of the chief factors can be summarised as follows :

(i) Availability—the question of relative availability of materials.
(ii) Processing—relative ease of production of the finished article.
(iii) Technical—general technical pros and cons.
(iv) Saleability—relative ease of selling, etc.
(v) Economics—questions of prices, costs and profits.

These are all interconnected. It is not within the province of this article to deal with them in detail as applied to cable sheathing ; one general point, however, is that taking all in all, if it is desired to find an alternative to lead for cable sheathing, aluminium is the most suitable on these five counts.

It is of interest here to note some published figures on the abundance of metals in the upper ten miles of the earth's crust.[1] Aluminium turns out to be easily the most abundant, the actual figure being 8·13%. The amount of lead is only 0·025% of that of aluminium, i.e. with aluminium reckoned as 100%. On the same basis copper is 0·12%. Other figures of interest are iron 62% and magnesium 26% on the same basis again. It is of interest to note the figure of 8% for titanium, a metal of increasing prominence and conceivably of importance in the future in cable applications.

Such figures do not tell anything like the full story, since the important sources are ore concentrations. They do, however, highlight the possibility that at some time in the future aluminium may be one of the few metals of general engineering importance abundantly available.

Published figures for actual world production in 1948 were, approximately, in thousands of short tons, 1,700 aluminium, 1,900 for lead. It would seem that at present the lead shortage fear would not appear to be a major factor, although this (and in fact recent actual shortages) has been one of the most important incentives for the development of aluminium for cable sheathing. It is perhaps more accurate to state that whilst, comparatively speaking, there is no dire lead shortage to-day the fear of such remains. Until recently the lead *stock* position was not good, owing to the high consumption as compared with production. Production, however, would now appear to have improved and world stocks and U.K. stocks would seem to be better, although fluctuating badly.

As regards aluminium, the chief world producers are at present the U.S.A. and Canada. Some figures, in short tons, for 1947 are as follows :

U.S.A.	449,000
Canada	270,000
U.K.	29,000
France	53,000
Norway	21,000
Switzerland	20,000

Pre-war, Germany and Japan were large producers, and large amounts are now produced in Russia. Much aluminium is imported by the U.K., largely from Canada. In 1947 for example we imported close on 96,000 tons from that country. It is of interest to give some very rough figures as regards U.K. production of fabricated products (they vary much from year to year).

	tons per annum
Sheet and strip	130,000
Castings	50,000
Forgings up to	40,000
Extrusions about	40,000
Of which Tubes amount to some ..	4,000
And Wire to some	3,000

The figures can be compared with a total annual lead consumption of the U.K. of approximately 300,000 tons, of which something approaching 100,000 tons is used for cable purposes. This includes lead for all such purposes, but assuming it to be mainly sheathing, it is equivalent to something of the order of 20,000 tons of aluminium, about five times the figure given above for the total extruded aluminium tube in the country. This figure indicates the order of magnitude of change of materials involved.

General Metallurgical Factors

Turning now to more purely metallurgical factors, the first question is that of availability of suitable qualities of material for sheathing. Practically the only ore of aluminium which is of major importance is bauxite ; much of the 8% of aluminium of the earth's crust is, in fact, not economic to exploit. The metal is obtained by the electrolysis of a molten salt bath consisting of a solution of about 5% pure alumina, prepared from the impure bauxite, in cryolite. The process is an expensive one, particularly so when the expense of refining the bauxite is taken into account. It is this, as well as technical difficulties, which limits the purity of the aluminium obtainable commercially. The maximum

[1] Metals—Heavy and Light," *Light Metals*, January 1950.

TABLE I.—COMPARATIVE PROPERTIES OF ANNEALED ALUMINIUM, LEAD AND COPPER.

Property	Pure Lead	Aluminium 99·99%	Aluminium 99·5%	Copper O.F.H.C.
Elastic Modulus lb./sq.in.	2×10^6	10×10^6	10×10^6	17×10^6
Tensile Strength tons/sq.in.	1·0	2·5	5·7	14·0
Yield Point (or 0·1% Proof Stress), tons/sq.in.	—	1·0	1·9	3·0
Elongation %	50	45	30	60
Brinell Hardness	3·5	15	20	42
Fatigue Limit tons/sq.in.	0·3	1·5	2·0	4·55
Stress to give creep rate of approx. 0·1% per year: lb./sq.in.	100	1,100	more than 4,000	1 ton/sq.in. gives creep rate of approx. only $0{\cdot}02 \times 10^{-6}$ in/in/hr at 150°C *i.e.* very small
Electrical Conductivity I.A.C.S.	V. approx. 8	64	61	102
Thermal Conductivity (C.G.S. Units)	0·082	0·57	0·53	0·92
Specific Gravity	11·34	2·70	2·70+	8·94
Electrode Potential (volts against hydrogen electrode) 20° C.	−0·13	−1·45	(approx. −1·45)	+0·35
Melting Point °C.	327	660	658·7	1,084
Coefficient of Thermal Expansion per °C.	29×10^{-6}	$23{\cdot}5 \times 10^{-6}$	Approx. same as previous col.	17×10^{-6}

purity in general is about 99·5%, the impurities ranging as a rule from 0·5 to 1%. With careful selection of materials purities of up to 99·8% can be obtained. Much of the Canadian metal imported has averaged over 99·7%. The production of aluminium of purity greater than 99·8%, however, requires special refining methods, which results in considerably increased cost. "Super-pure" metal of purity greater than 99·95% is regularly produced, but its cost is much greater than that of the ordinary commercial metal.

Compositions of aluminium and alloys for tubes for general engineering purposes have been specified in B.S. 1471 : 1949, one of a series dealing with wrought products. The aluminium qualities specified are :

T 1A not less than 99·8% pure
T 1B " 99·5% "
T 1C " 99·0% "

The two chief impurities which occur in commercial aluminium are silicon and iron. Specification T 1B gives as maxima for these 0·3% and 0·4% respectively. Some discussion of the effect of impurities on aluminium properties will be given below.

It will be noted that in regard to the purity of commercial metal there is a substantial difference between aluminium and lead. Much of the commercially obtainable pure lead is of very high purity, and the cable sheathing specification B.S. 801 gives 0·1% as maximum total impurity for pure lead cable sheaths. Commercial lead in fact ranges generally from about 99·75% to 99·95% pure, whilst 99·99% is by no means uncommon.

The fundamental crystalline structures of pure lead and aluminium are basically the same, both having a face-centred cubic structure, which is one of the three common "crystal lattice" structures of metals. This structure is one that is characterised by a high degree of ductility and ease of working.

It may be noted that the grain size of lead cable sheaths varies from about 1 mm. mean diameter (0·7 mm. a desired maximum) for the pure metal to about 0·2 mm. for alloys. The grain size of extruded aluminium tube (99·7% pure) is of the order of 0·1 to 0·15 mm. mean diameter but can be considerably less. There are large variations in these figures. In this connection there is another point of interest and relevance. In lead, particularly pure lead and dilute alloy products, the occurrence of adjacent zones of large and small grains is not uncommon, and their presence can lead to undesirable forms of weakness. "Zoning" is also not very desirable in aluminium, but is less liable to occur and less dangerous than for lead in extruded aluminium tubing as used for cable sheath. Another important comparison between lead and aluminium concerns ease of recrystallisation and grain growth. For pure lead, recrystallisation can take place at room temperature, whilst grain growth can occur at the temperature of 70° C. which cable sheaths sometimes reach in operation. In super-pure aluminium the recrystallisation temperature of the cold-worked metal can be less than 100° C., but the temperature is greatly affected by the presence of impurities in commercial aluminium. In 99·5% pure aluminium the recrystallisation temperature of the cold-worked metal is about 300° C. Extensive grain growth does not occur in general until temperatures substantially greater than this are reached. Consequently, extruded and drawn aluminium sheathing is not likely greatly to alter its properties at cable sheath operating temperatures.

Some comparative properties of interest for aluminium, lead and copper are given in Table I. Most of these properties, it should be noted, have considerable variations and the figures given are, in general, approximations. The properties given apply to the materials in substantially the fully annealed condition. Aluminium and copper can be much hardened by processes of cold work ; pure lead is one of the few metals not hardened by such treatment.

It will be seen that the mechanical properties of aluminium are, in several respects, intermediate between those of lead and copper. When one takes into account the strength-to-weight ratio, the values compare well with those for copper. From the present point of view, the much superior tensile strength, fatigue limit (resistance to vibration) and creep resistance (resistance to gradual deformation under small stresses) of aluminium as compared with lead are of particular importance. These values can, of course, be considerably increased for lead by the addition of alloying agents, but there are few cable sheath alloys which have values better than double those given for lead. The large effect of the presence of 0·5% impurity in aluminium is clearly shown.

Another comparison of interest in certain connections is that of electrical conductivity. That of aluminium compares very favourably with copper, from which arises the use of aluminium for electrical conductors. The conductivity of aluminium is about eight times that of lead, a factor of some importance in connection with losses in single-core cables, which are higher for bonded aluminium sheathed cables than for lead. Addition of alloying elements decreases the electrical conductivity of the metal, but, for feasible alloys, not to a sufficient degree to make a great deal of difference. Some comparisons of interest are given in Table II, which lists some specific resistance values. Values of proof stress and ductility are also shown. It is worth noting that proof stresses of as much as 40 tons/sq.in. are obtainable in some heat-treatable aluminium alloys.

Several aluminium alloys can be obtained in the form of extruded tube, but few are of great interest for cable sheathing. Nearly all require higher extrusion temperatures than the unalloyed metal, and on the whole the disadvantages outweigh the advantages for normal purposes. The $1\frac{1}{4}$% manganese alloy (D.T.D. 213A, etc.)

is of interest as one in very general use in the form of sheet, tube and formed articles.

In connection with special alloys, it may be of interest to mention a U.S. installation of a gas pipe line, in aluminium alloy, 18 miles long, in Alabama. The dimensions are $8\frac{5}{8}$ in. o.d. × $\frac{1}{4}$ in. wall, and the working pressure is 200 lb./sq.in. The alloy used is 63S, in the extruded and heat-treated condition. The nominal composition is 0·4% silicon and 0·7% magnesium. The pipeline was laid in 40-ft. lengths and heliarc butt-welded. A material known as No-Oxid pipe-wrap has been used for protection. The bursting pressure of the pipe is given as 1,750 lb./sq.in.

Methods of Sheathing

The method of sheathing which is at present used on a production scale is the so-called " dieing-down " process. It can be said at once that although this method has been developed to a high degree of efficiency and meets present needs, it by no means represents finality, and the future may well show that direct methods of sheathing will take its place on large-scale production.

Some of the chief methods possible for aluminium sheathing can be listed as follows :

1. Extrusion directly on cable.
2. Sinking-down of oversize tube (dieing-down).
3. Forming of strip around cable and welding by—
   (*a*) Resistance welding.
   (*b*) Cold welding.
   (*c*) Argonarc welding.
4. Special methods, e.g. Alpeth, Hackethal processes.

Extrusion

The chief difficulty in the extrusion of aluminium cable sheath directly on to cable is the high temperature required, resulting in damage to the core, whilst the pressure requirements are also considerably greater than for lead.

The extrusion of lead cable sheathing requires a temperature of the order of 250° C., aluminium tube may require about 450° C. (copper nearly 900° C.). It is perhaps of interest to mention impact extrusion, by which a tube can be formed by the sharp blow of a punch on a slug of metal standing in a shallow die, the metal being forced up over the punch by the sudden pressure. The method is largely used for the manufacture of collapsible tubes and the like from tin, lead or aluminium, and the process is carried out without application of heat. There would appear to be no possibility, however, of such a process, which produces only short tube lengths, being used for cable sheathing.

Before proceeding to particular consideration of the problems involved in the extrusion of aluminium tube or sheath, it will be as well to deal briefly with the basic

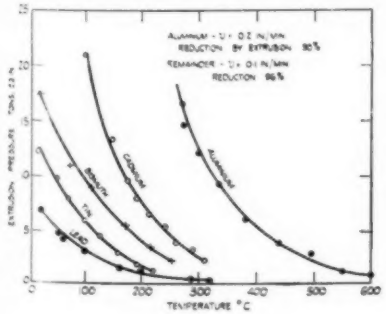


Fig. 1. — The effect of temperature on the pressure of extrusion.

TABLE II.—PROPERTIES OF SOME ALUMINIUM ALLOYS.

Material	Condition	Specific Resistance microhm/cm.cube	0·1% Proof Stress tons sq.in.	Elongation %
Superpure Al	Annealed	2·68	1·0	45
99·5% Al	Wrought annealed	2·80	1·9	30
	,, half-hard	2·82	6·2	15
	,, hard	2·85	7·8	10
99·0% Al	,, annealed	2·87	2·0	45
	,, hard	2·89	8·5	10
	Extruded	2·86	3·5	27
0·5% Mg.	Extruded and solution-treated	3·5	6·5	30
	Aged	3·3	12	20
1·5% Mn. (D.T.D. 213A)	Wrought annealed	3·4	3·7	25
	,, half-hard	4·2	7·0	10
	,, hard	4·3	10	5
2% Mg. & 0·25% Mn. (e.g. Noral M578)	Wrought annealed	4·6	6	20
	,, three-quarter hard	4·9	14	4
12% Silicon	Cast	4·7	4·5	7
Copper	High conductivity	1·55	3	60
Lead	Pure	19	—	—

factors involved in any method of metal extrusion. The pressure required for extrusion depends upon the " resistance to deformation " of the metal, the temperature, the extrusion ratio, the speed of extrusion, frictional losses involved in the metal flow and the precise nature of the flow. Considerable work has been done of both a fundamental and practical nature on the precise relationship between these quantities. The resistance to deformation of the metal, mentioned above, is a quantity which can be measured, but of which, in fact, quite inadequate measurements in most cases exist. It is largely the value of the resistance to deformation which determines the minimum temperature at which any given extrusion process can be carried out. Fig. 1 shows some published values for the relationship between extrusion temperature and pressure for lead, tin, bismuth, cadmium and aluminium.[2] It will be seen that much higher temperatures and pressures are required for aluminium than for lead. It may be noted also that the pressures required for the extrusion of aluminium in practice are, in general, considerably greater than those shown in this figure, the values being from small-scale experiments. Results from some of our experiments on the comparison of aluminium (99·7% purity) and lead in small-scale extrusion are shown in Fig. 2. The values of resistance to deformation shown have been derived from actual extrusion trials by use of the logarithmic relationship between pressure and extrusion ratio. The derived values are probably too high, but of the right relative order. It will be seen that the values for aluminium are two to three times those of lead, and there is a more marked dependence on extrusion speed. The extrusion ratio used in practice for commercially pure aluminium depends upon the capacity of the press, the temperature and speed of extrusion and the form of the product, and a figure of about 20 : 1 is not unusual. A smaller ratio must be used for alloys than for pure metal as these are harder to extrude and require higher pressures. In tube production higher ratios may be used.

The flow of the metal in even the simplest form of extrusion is quite complex, and no attempt will be made to deal with this question in detail here. Non-uniform flow, if certain precautions are not taken, can lead to defects in the extruded product. It is also of importance in connection with one of the problems under investigation, the joining up of successive billets in the press, and this will now be dealt with.

2 C. E. Pearson " Extrusion of Metals," London, 1944, p. 132.

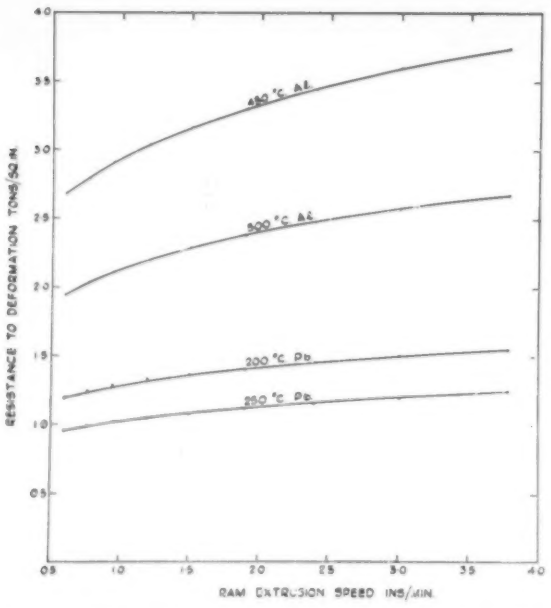


Fig. 2.—Comparison of the resistance to deformation of aluminium and lead.

In order to obtain the very long lengths of tubing required for cable sheathing it is necessary either to start with a large billet, or to use a succession of small billets, which must join up during extrusion. In the extrusion of lead this presents a considerably simpler problem than is the case for aluminium. The lead can be poured into the press container in the molten condition, being allowed to solidify before extrusion proceeds. Successive charges are poured in one after the other and joining up takes place by the melting of the surface of the solid metal by the new liquid metal. Even so, great precautions are necessary to ensure a product free from defects. In the extrusion of aluminium, solid pre-cast billets are used, owing to the high temperature required for the use of molten metal charges, the attack on the steel of the container by the molten metal, and other metallurgical and engineering difficulties. It is a considerably more difficult problem to join up a succession of solid billets, the main sources of difficulty being as follows:

(i) The entrapment of air between the successive billets.

(ii) Non-uniformity of flow leads to the presence of cavities and the entrapment of the oxide skin of the original billet in the slug left when it has been extruded. It is in fact usual to remove and discard this slug before extruding a fresh billet in ordinary practice.

It will be seen that in order to obtain sufficiently long lengths, 200 yards or so, of tubing of large diameter from single billets, very large presses are required, up to 3,000 to 6,000 tons capacity, and capable of dealing with billets of up to 20 in. diameter. Among several methods in which the problem of billet joining is being dealt with may be mentioned the use of reject ports (Pat. App. No. 16817/50). The idea is to reject metal as may be required through ports at the front end of the wider part, with the hope of getting rid of unsound metal and favourably modifying the flow.

Before proceeding to deal with the problem of the direct sheathing of cable by aluminium, it may be well to recall that there are two main types of press used for tube extrusion (i) the mandrel press and (ii) the bridge or porthole die press. In the former, a central mandrel passes through the press container and the metal is extruded as tube through the annular space between this and the die. A hollow billet must be used, with the central portion accurately bored out to fit over the mandrel, alternatively the billet is pierced in the press. In the second method, the central mandrel is dispensed with

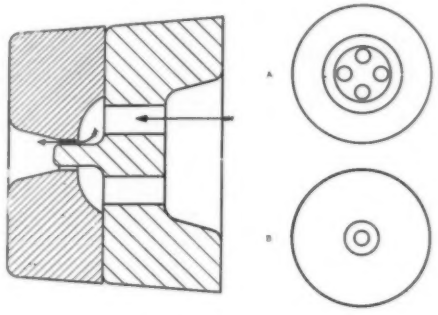


Fig. 3.—Diagram of ante-chamber die (A—back view; B—front view).

and the billet is solid. Fig. 3, shows one design of die used.[3] The metal from the container passes through the portholes, as shown by the arrow, and is extruded as tube through the annular space around a short internal mandrel projecting into the die. It will be noted that the metal is split up into separate streams as it passes through the portholes and has to reunite in the die space. Thus, if four portholes are used (the number used depends upon the size of die) four welds (seams) are required for the metal to reunite. The question of the quality of tubing produced by this method will be dealt with below; suffice it to say here that with proper precautions no difficulty is experienced in obtaining sound tubing. In the best quality of tubing, particularly from aluminium of high purity, the "seams" can hardly be detected. The tubing is in fact always referred to as "seamless" since no artificial seam is made. All the tubing we are at present using for sheathing is produced by this method. Aluminium extrusion presses can be vertical or horizontal, the larger sizes being usually horizontal, but they are invariably of the "straight through" type, that is the extrusion is in direct line with the ram and container and not at right angles to it as in an ordinary lead sheathing press. It may be noted that the aluminium billets must be pre-heated to the required temperature and the press container also heated, usually electrically.

Extrusion Directly on to Cable

This is of course ordinary practice with lead. For aluminium there are considerable difficulties, chiefly due to the higher temperatures required, resulting in deterioration of the paper dielectric and surrounding tapes of the cable core as it passes through the press. Ordinary impregnated paper cables will commence to char rather rapidly at about 350° C. They commence to deteriorate at temperatures below 300° C. and will deteriorate ex-

3 C. Smith, *Journal of the Institute of Metals*, January 1950, **76**, 449.

tremely rapidly at ordinary aluminium extrusion temperatures.

In order to solve this problem one or more of the following conditions are necessary :

(i) The use of low extrusion temperatures, preferably below 350° C., and if possible 300° C. or even lower than this.

(ii) Keeping the core cool as it passes through the press, e.g. by water cooling of the hollow mandrel through which it passes.

(iii) Keeping the length of the passage of the core in the press as short as possible.

(iv) Extruding at high speed so as to keep the time during which the core is exposed to the high temperature as short as possible.

(v) Protection of the core, e.g. by thin asbestos or other heat-resisting wrappings.

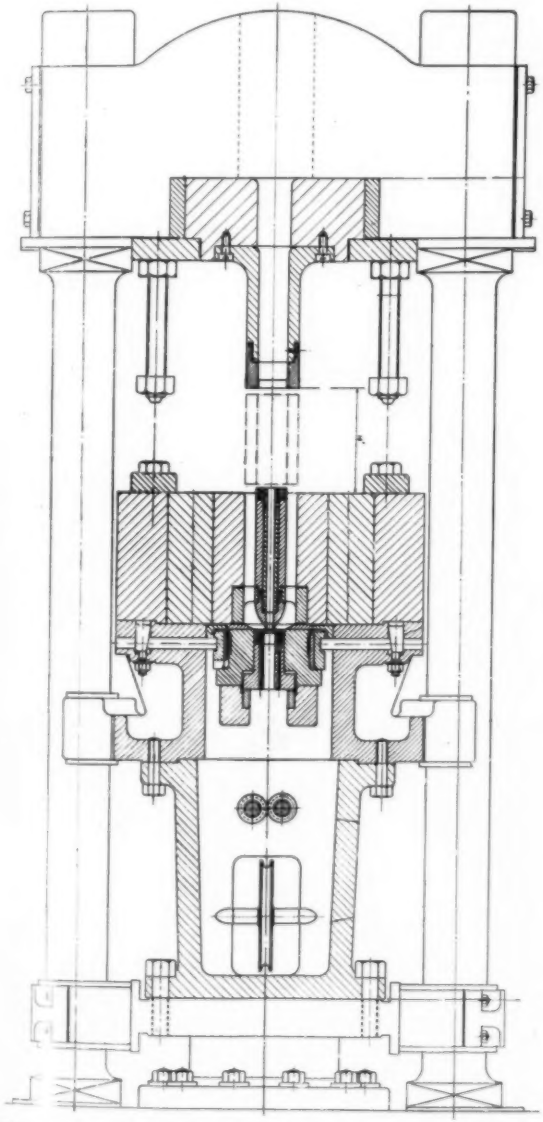
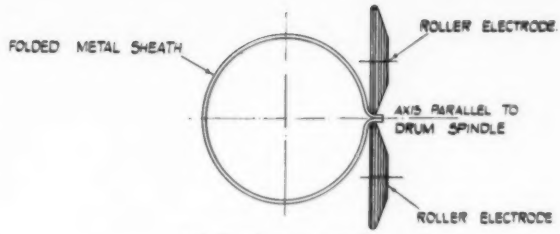
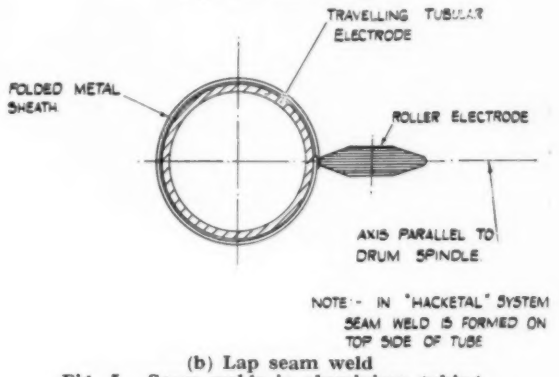


Fig. 4.—Vertical lead-sheathing press converted for experimental work with aluminium.



(a) Bent seam weld.


TRAVELLING TUBULAR ELECTRODE
FOLDED METAL SHEATH
ROLLER ELECTRODE
AXIS PARALLEL TO DRUM SPINDLE
NOTE :- IN "HACKETAL" SYSTEM SEAM WELD IS FORMED ON TOP SIDE OF TUBE

(b) Lap seam weld

Fig. 5.—Seam welds in aluminium tubing.

(vi) The development of dielectrics of superior heat-resisting properties.

(vii) Extruding tube oversize, followed by drawing down afterwards.

Many experiments have been carried out on the direct extrusion of aluminium on to cable. A very good account of some pre-war German work, claimed to have achieved considerable success, has been given in *Light Metals*.[4] The maximum diameter of sheath obtained would appear to be about 1 in. Split billets had to be used, pushed in over the mandrel. It was claimed also that good joins were obtained with successive billets. In order to obtain a sufficiently low extrusion temperature superpure aluminium had to be used, and it was claimed that satisfactory extrusion could be achieved at temperatures as low as 280° C.

Fig. 4 shows a converted vertical lead sheathing press of 2,000 tons capacity fitted for experimental work on the vertical extrusion of certain types of aluminium sheathing. The press will extrude up to about 1 in. diameter sheath.

Welding of Formed Strip

A method other than direct sheathing or drawing into tube likely to be applicable on a large scale is the welding of pre-formed strip. The strip is formed into tube, while the cable is fed in, by the use of a suitably designed roller machine. The two ends of the strip can form a fin or lap. These are shown in Fig. 5. For subsequent welding, the fin is the easiest, but the bending properties of the tube are bad. The bending properties of these welded sheaths can present severe problems.

Small size welded tubes in both stainless steel and aluminium have, however, been successfully produced.

For welding, three methods are possible :

(i) Electric resistance welding.

(ii) Pressure welding.

(iii) Argonare welding.

4 " Cable Sheathing in Aluminium," *Light Metals*, September 1946.

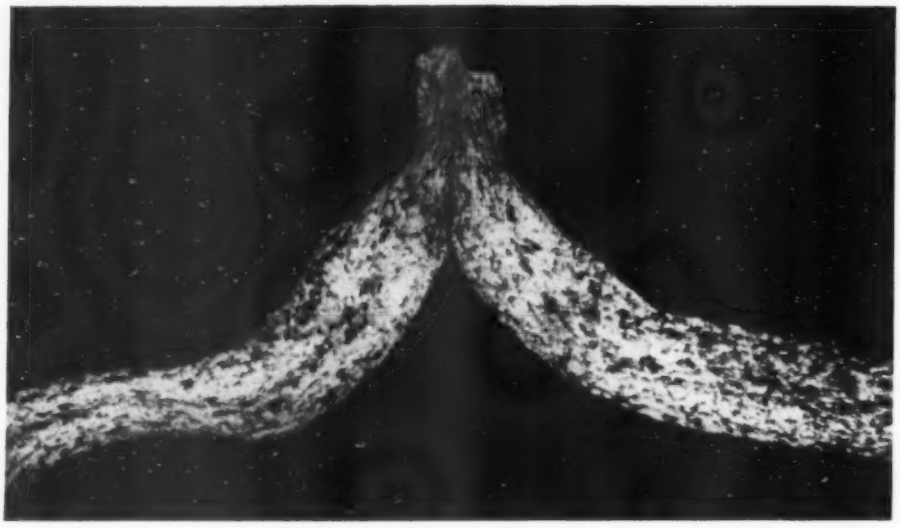


Fig. 6.—Structure of cold weld ×21

(i) *Resistance welding.* The electrodes are applied as shown in Fig. 5*a*, the fin passing between a pair of roller electrodes, and in 5*b*, a reciprocating action being required for the inner electrode when making a lap. Resistance welding is readily applicable to steel and stainless steel sheath (of small diameter) has been produced in this way.

(ii) *Pressure Welding.* Various metals can be joined by exerting a high localised pressure, when welding takes place at the interface. When carried out in the cold (cold welding) a high deformation is usually required, and another difficulty is the hardening of the metal and resultant embrittlement. The process can be carried out using a similar arrangement as for resistance welding, the wheels, instead of acting as electrodes, apply a high pressure on each side of the fin. The strip edges are cleaned by high-speed scratch brushing immediately prior to forming the tube. The structure of a cold weld in an aluminium tube is shown in Fig. 6. The process has received detailed study, but its applicability would seem to be limited.

(iii) *Argonarc Welding.* This method consists of striking an arc between the fin and an electrode (tungsten commonly used) to cause the two edges of the fin to fuse together. A stream of argon gas passes around the electrode tip and over the arc and the use of this inert gas shield obviates the need for a flux, such as required in ordinary arc welding to clean the metal. The heliarc method is a similar method using helium gas, chiefly used in the United States. The structure of an argonarc fin weld in aluminium is shown in Fig. 7.

Another development in argonarc welding is the use of a butt-type of weld, the two edges being brought together without a fin.

The Hackethal process is an attempt at overcoming the bending difficulty by corrugation of the tube, but requires a lap weld.

The Alpeth process, developed in the United States, makes use of a corrugated aluminium tube, formed by wrapping pre-corrugated strip around cable core and over the metal is an extruded polyethylene covering. Good mechanical and bending properties and high resistance to moisture penetration are claimed with this construction.

The Tube-Sinking Method

The method of aluminium sheathing at present in production is that of drawing the cable into a long length of tube, into which it can be easily pulled by allowing sufficient clearance. The tube is then passed through a die to decrease its diameter to a close fit on the cable. For solid-type cable an exact fit is aimed at; with gas-filled cable a small clearance is left for gas space. A difficulty in this process is that the tubing has to be in a straight length, or have no more than one or two very large radius bends, for the core to be drawn in.

The basic factors concerned in such a process are:

(i) The amount of diameter reduction which can be carried out—the drawability as it may be called.
(ii) The degree of work hardening, *i.e.* increase in hardness which results from the process.
(iii) The dimensional changes, *i.e.* alterations in length and wall thickness which accompany the diameter decrease.
(iv) The force required to pull the tube through the die.

As regards the question of drawability, it may first of all be recognised that this process differs in degree rather than in kind from other metal drawing processes. For all such processes there is a theoretical maximum for drawability, any attempt to exceed which will result in breakage of the wire, tube, etc. This maximum corresponds to about 50% reduction in cross-section for aluminium. The process with which we are here concerned, which has come to be referred to as "dieing-down," is a hollow tube-sinking process, in which there is small alteration in wall thickness. It follows that the

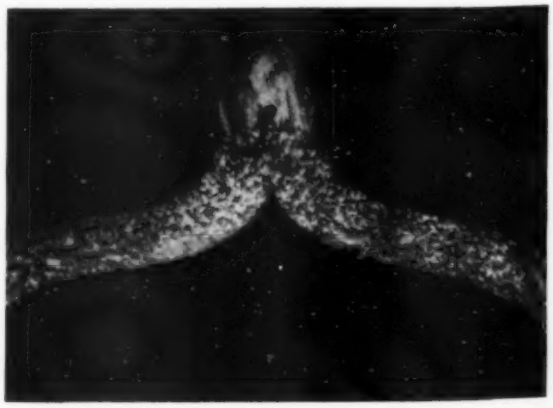


Fig. 7.—Structure of argon-arc weld ×9

maximum reduction which can be applied is about 50% decrease of diameter, in one pass. In practice, reductions of diameter less than this are generally concerned in sheathing, a common figure being of the order of 10 to 20%.

With regard to work-hardening, aluminium, like most metals (lead is an exception) becomes harder and more brittle when it is drawn, etc. in the cold. This results in the aluminium sheath being considerably more springy and harder than the tube from which it is made.

The dimensional changes of the aluminium tube resulting from its decrease in diameter in the sinking process follow from two facts—(*a*) the density is not altered, (*b*) the inside of the tube is not supported on a mandrel. From (*a*) it follows that the increase in length is commensurate with the decrease in diameter. In fact if d_0=initial diameter of tube, l_0 its initial length and d_1, l_1 the corresponding values after drawing through the die then :—$d_1 l_1 = d_0 l_0$, if the wall thickness is unaltered, *i.e.* the tube lengthens proportionately as the decrease of its diameter. As regards (*b*) the result is that the wall of the tube actually thickens a little owing to the circumferential compression in the tube as it passes through the die. This must be allowed for in sheathing cable, but is rarely greater than one or two mils.

The pull required for tube sinking depends chiefly upon the degree of reduction, the design of the die and the lubricant used. In practice, values from a few hundredweight to about one ton are needed. The design of the die considerably affects the exact final diameter of the tube ; a die with a large taper angle will result in a tube of considerably smaller diameter than the diameter of the die orifice itself ; a small angle taper leads to closer correspondence, but higher loads and thicker dies are then required.

Detailed analyses of the stresses and strains in tube sinking have been given by Hill[5] and by Chung[6]. The results indicate that with, for example, a die semi-angle of about 10° and a value of coefficient of friction of, say, about 0·1, the drawing stress required for about 15% sink is about 25% of the yield stress of the material, which is rather smaller than obtains in practice. The pulling load, it may be noted, is given by the product of the drawing stress, the final material cross-section of the tube, and the cosine of the die semi-angle. They also indicate for the same conditions, a theoretical wall-thickening of about 7% which is somewhat greater than observed in practice. It is of interest to note that a maximum wall thickening is indicated at about 30 to 40% sink, and the effect then rapidly decreases.

To be continued

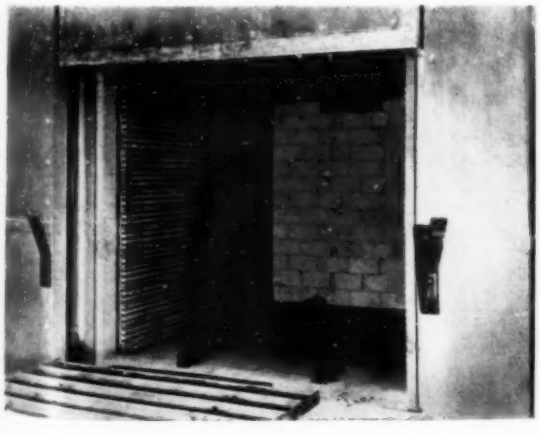
5 R. Hill " Mathematical Theory of Plasticity," Oxford, 1950, p. 269.
6 S. Y. Chung, *Metallurgia*, 1951, **43** (259), 215.

Stress-Relieving Furnace

AN interesting electric furnace, designed for stress-relieving welded steel frames after fabrication and capable of operation at temperatures up to 650° C., has recently been supplied to the Admiralty by G.W.B. Electric Furnaces, Ltd., of Dudley. The interior usable dimensions are 6 ft. long × 4 ft. wide × 4 ft. high, and the heating chamber is fitted with two rows of heat-resisting cast-iron rollers. As can be seen in Fig. 1, one row of rollers is flanged to locate the charge carrier while the other row is plain to accommodate expansion.

Since the normal operating temperatures are in the range where convection constitutes the prime factor in heat transfer, two air circulating fans (visible in Fig. 1) are fitted under the chamber roof. Each fan is driven by a 1 h.p. electric motor with an extended spindle forming,

Fig. 1



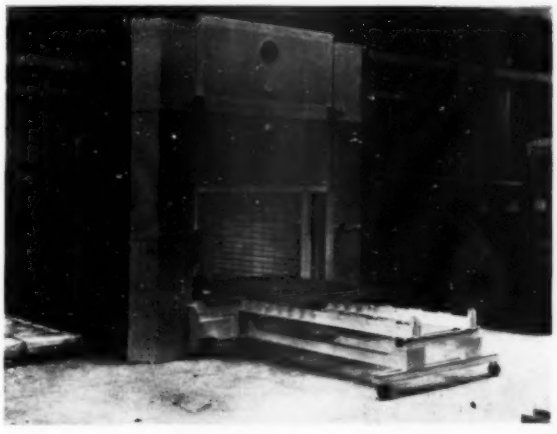


Fig. 2

in effect, the fan shaft. Following standard practice the rating of 46 kW's is balanced over a three-phase supply and in this particular instance is reducible to approximately one-third of maximum by means of a hand-operated Star/Delta switch. Heating elements of 80/20 nickel-chromium alloy wire in coil form are fitted in the sidewalls, roof and hearth of the working chamber, supported in specially moulded refractories. Temperature control is effected by means of a Kent two-point recording controller working through solenoid operated switchgear, and an oil circuit breaker.

A feature of this installation is the mobile, castor-mounted loading table seen in Fig. 2. This can be bolted to the front of the furnace to suit the circumstances and is fitted with two rows of idler rollers corresponding to those fitted in furnace hearth.

Durable Enamel Finishes on Zinc-Base Die-Castings

By E. E. Halls

Metal shortages have given impetus to the development of processes for producing an enamelled finish on zinc-base die-castings in place of the electroplated coatings which were formerly applied. In this article the author discusses the properties of enamelled finishes, paying particular attention to the pre-treatment necessary.

IN the post-war period, engineering industries have designed for zinc-base die-castings, and in consequence the service efficiency of the latter has probably been greater than ever before. Advantage has been taken of the reputable alloys to die-cast components that would otherwise occupy valuable machining capacity and result in much scrap metal. The low die-casting temperature of zinc gives high outputs from the tools and dimensional accuracy is exceedingly good. It is noteworthy, too, that the surface finish of components from experienced die-casters is exceedingly good, showing that improvement much desired in the metal finishing shop. X-ray examination has been exploited at the tool try-out stages to eliminate major porosity or to ensure that it occurs in sections where it is not objectionable, and where it cannot lead to stress failures or give trouble in the polishing shop.

Zinc-base die-castings have particularly been exploited in the domestic apparatus and appliance industries, i.e., on radios, refrigerators, electrical apparatus and vacuum cleaners, and on automobiles and car accessories. In many of these applications a bright finish of permanent character is highly desirable. Nickel-chromium electroplate has been employed with considerable success, and improvements in electroplating processes have assisted. The bright nickel electrolytes and, in America, the fluoride-containing chromium plating solution, have been valuable in this direction.

Enamel Finishes to Replace Electroplating

Zinc castings are not easy alloys upon which to deposit sound electroplate coatings. The casting needs to be dense and of good surface condition and, preferably, designed to be free from sharp edges, re-entrant angles, recesses and blind holes. Surface preparation by cleaning and acid etching has to be closely controlled, a preliminary coating of copper to a controlled thickness must be applied from a special electrolyte, and the nickel and chromium processes also have to be closely controlled. The plated coatings tend to develop tiny blisters in service if all these conditions are not properly satisfied: this blistering is still encountered in practice and, in fact, is the most important problem in the practical plating of miscellaneous components in these alloys.

To-day there is not the available nickel for indiscriminate electroplating of die-castings, however desirable such a finish may be. In fact, the indications for some time have been that other more important applications will consume the limited world production of this metal, and the time when it will again become freely available for electroplating cannot be foreseen. This leads to the consideration of enamel finishes as an alternative, and it is probably true to say that in the majority of cases an enamel finish to match, tone or contrast with the finish of the background to which the casting is assembled, can be satisfactorily chosen.

In applying a paint, lacquer or enamel finish, it has to be remembered that zinc is a very reactive metal, and that it will combine with fatty acids in the oil or resin of the paint film to form a soap, causing the finish to fail. This is evidenced by blistering and/or softening of the finish, which can then be scraped off with the finger nail. In some cases, this occurs in a matter of weeks or, at the most, months. This problem can be fully taken care of by chemical pre-treatment of the surface; phosphate processes are the most reliable for this purpose, although selected chromate treatments can also be used satisfactorily.

In addition to chemical pre-treatment for assuring durable organic coatings upon zinc castings, there is to-day a special primer, generally known as "etch" or "wash" primer, that fulfils the same function. The use of this represents perhaps the most outstanding advance in industrial enamel finishing since the war. The material may be sprayed and air-dried or stoved and then followed by a conventional enamel type, i.e., air-drying or stoving, cellulose, oil or synthetic base, with every satisfaction. Its adoption means that "wet" processes can be avoided. It is only neceesary to clean the castings, and this may be done by solvent vapour process if no polishing or other contamination has occurred, or by liquor-vapour method in the latter case, employing the orthodox trichlorethylene plant.

There is no finish in the paint or enamel range that can be substituted for an electroplate coating to give the high lustre, reflectivity and smoothness of, for example, nickel/chromium. Nor is there one to provide electrical conductivity. When these properties are not essentials, it is practicable to select an organic finish that is simple and economic to apply, that provides good durability and protection, and that can provide for decorative or artistic effects.

In its simplest and most economic form, the organic finish may be the chemical pre-treatment, or the etch primer, and a single finishing coat, or the latter may be a double coat to ensure maximum impermeabilty to moisture and corrosive influences. Again, it may utilise the decorative enamels such as "cracking" or "shrivel," or the metal powder finishes such as silver or gold, or it may use an enamel coat followed by a clear varnish, colourless or tinted. The enamel type may be cellulose, urea- or melamine-modified alkyd, oil-modified alkyd, etc., so that the facilities in existing finishing shops with respect to drying or stoving arrangements can be utilised.

At this juncture, it must be stressed that the type of organic medium, and the quality of the particular product selected, contribute materially to the serviceability and long-period durability of the finish. For example, cellulose finishes gradually age to hard brittle films. Where the coating is regularly washed and wax polished, e.g., on an automobile finish, it has a long life, but on miscellaneaus components where this maintenance is not practicable—and this probably represents the majority of cases—then cellulose finishes in time have poor resistance to mechanical stresses such as impact shock or vibration. Although proper pre-treatment of the castings may go a long way towards suppressing or delaying such deterioration, the tendency to degradation is always present. Stoved finishes always give best performance, the urea- or melamine-modified alkyds for hardness of film, best retention of colour and resistance to ulta violet light, and the oil-modified alkyds for good durability combined with flexibility. Further, the quality of the pigment and obliterating filler is important. Thus, a white, light grey or other pale shade having chalk as the main extender will not possess the exposure resistance of an enamel in which titanium white is used for this purpose. Likewise magnesia as a flatting agent is too reactive a base in humid and slightly acidic atmospheres to give the same performance as a more inert material such as silica flour or china clay. Many of these features are compositional details beyond the scope of the user's facilities for assessment, but they do emphasize the need for careful selection. It is almost imperative that the latter be backed by thorough laboratory testing, including exposure trials, and that some observations should be made on service performance. Price alone is not always a good guide, although low initial cost per gallon does usually signify that less expensive ingredients have been utilised in production.

Chemical Pre-treatment

Phosphate Methods.—The assured chemical pre-treatment processes for zinc are those of the phosphate type. These can be broadly classified into two groups, *viz.*, the full or long period processes, and the short period types. Normally proprietary processes are employed, although the user may fairly readily prepare his own solutions if he so desires. All of the processes have to be controlled with respect to time and temperature, although not critically, and of course, the work must be clean before treatment. The solutions also have to be maintained to compensate for consumption of chemicals in the actual reaction, also for evaporation losses. Chemical control is by very simple alkalimetric titration, from which the quantity of replenishment chemicals is calculated.

In a typical full period process, the work is immersed for 10 to 15 minutes at 210° F. nominal temperature. The work should not be packed tightly but freely spaced to allow circulation of solution to all surfaces. After treatment it is washed in hot water at about 180° F. (two rinses in separate waters being preferred) and dried off. Oven drying above the boiling point of water (220 to 350° F.) is advocated, as this ensures a dry surface, and particularly drying-out in recesses, before enamelling. However, oven drying is not essential.

The short period processes use the rapid phosphate treatments designed for steel as well as for zinc. The immersion period is from 3 to 5 minutes and the temperature of the order of 180° F. plus or minus 5° F. The second after-wash in this process for steel usually contains a little chromic acid, of the order of 0·5 lb./100 gall., and this may be retained for the zinc work. Oven baking of the treated work is again advised.

The full phosphate process converts the zinc surface into an inert porous, phosphate film, dark grey in colour. It is highly absorbent, thereby giving a good key to paint but, being inert, it does not react chemically with the fatty or resinic acids in the paint film.

The short phosphate process gives an almost imperceptible film, usually shown by dulling or slight greyness of the surface, but still providing a very good base for painting or enamelling. The resultant finish has not the same durability as that provided by the full process but it suffices for most needs. Further, it is advantageous where certain areas of the zinc casting have to be left unenamelled, because the full process treatment, being so porous, exhibits, under severely corrosive conditions, an apparent greater degree of superficial deterioration.

Chromate methods.—The chromate processes are represented by the well-known chromate passivation process of specification D.T.D. 923. This confers appreciable improvement to the adhesion of cellulose finishes but, in general, very little to stoved enamels. The probable explanation is that the chromate films produced are essentially hydrated chromates, and dehydration occurs during stoving, with adverse influence upon the adhesion of the coating. This can be overcome by carefully selecting process conditions, and a very satisfactory procedure uses a relatively acid solution as follows :—

The complete sequence of operations must include an aqueous alkaline degreasing, followed by water washing, and a very dilute sulphuric acid rinsing (1 part sulphuric acid in 1,000 parts of water) to ensure no carry-over of alkali into the chromate solution, followed by the actual chromate immersion. The solution for this is maintained at 2 lb. of sodium bichromate and $2\frac{3}{4}$ oz. of sulphuric acid per gallon. Work is immersed for 10 seconds at room temperature, keeping the temperature as near 70° F. as possible. Work is briefly washed and allowed to dry. Two rinse solutions are used, with an in-and-out immersion in the first to remove the bulk of the chromating chemicals, and a few seconds in the next with good agitation. The ideal wash is that which *just* removes all the chromate solution. The wash waters are used at room temperature. Drying off is likewise at air temperature but circulated air may be used. The parts should be allowed at least 16 hours air drying before finishing.

In this sequence of processes, work must be well drained in passing from one operation to the next, but not allowed to become partially dry. One of the best alkalis for pre-cleaning comprises 8 oz. each of caustic soda and sodium fluoride per gallon, operated at 170° F., and with immersion for 15 to 30 seconds. An alternative uses 6 oz. of tri-sodium phosphate per gallon, short of boiling, with 10 to 15 minutes immersion, or 1 minute if used cathodically at 50 to 100 amp./sq. ft.

The chromate treatment is not so suitable for commercial production as the phosphate processes, because of the critical operating conditions. It is, therefore, used only in specialised circumstances. One of the latter is when electrical contact is required, phosphate being relatively insulating, whereas the chromated surfaces

TABLE I.—BEHAVIOUR OF CHEMICALLY-TREATED DIE-CAST ZINC SURFACES UNDER EXPOSURE TEST CONDITIONS.

Test Conditions	Chemical Pre-treatment			
	None	Full Phosphate	Short Phosphate	Chromate
Salt Spray	Gradual white corrosion, fairly heavy in 4 weeks.	White corrosion, slight at first, fairly heavy in 4 weeks.	Only slight white corrosion in 4 weeks.	Unaffected in 4 weeks.
Mild Warm Humidity.	A few white corrosion spots in 30 weeks.	Gradual slight whitening, condition poor in 30 weeks.	A few white corrosion spots only in 30 weeks.	Unaffected in 30 weeks.
Open Atmosphere.	A few white corrosion spots evident in 4 weeks, only little further deterioration in 30 weeks.	White corrosion spots in 4 weeks and rather pitted in 30 weeks.	Slight corrosion in 4 weeks, little further degradation in 30 weeks.	Unaffected in 4 weeks and practically unaffected in 30 weeks.
Severe Hot Humidity.	Immediate development of white corrosion, exceedingly heavy in 20 days.	Gradual development of white corrosion, very heavy in 20 days.	Only very slight white corrosion, in 20 days.	Virtually unaffected in 20 days.

are good conductors, and maintain low contact resistance even under damp exposure conditions.

Exposure Test Conditions

As it is deemed of interest, a comparison of the chemically treated zinc surfaces under corroding conditions is presented from exposure test results in Table I. The tests are the same as used for enamelled specimens discussed later, and comprise the following.

Salt Spray Test.—Specimens suspended in the mist produced by atomising 20% salt solution with clean, moist compressed air at room temperature. One day's test comprised 8 hours with the atomiser operating, the specimens being left in the mist laden atmosphere for the remainder of the day. They were washed in running water and dried on a soft cloth daily.

Mild Warm Humidity Test.—Exposure of the samples to daily cycles of 70% humidity at 55 to 60° C., during the 8-hour day, cooling off to atmospheric temperature with rise in humidity to 100% and condensation for the remaining 16 hours.

Open Atmosphere Exposure.—Open weather exposure without any protection from direct rain, etc., by suspending from a frame, facing S.W. in a mild industrial or suburban atmosphere.

Severe Hot Humidity.—Subjection of specimens to 100% humidity, maintained throughout daily cycles of 16 hours at 70° C. and 8 hours cooling off with condensation to room temperature.

Discussion of Results

The points enumerated above for unenamelled castings are confirmed by the test evidence. The full phosphate coating does not impart protection under any of the test conditions, in fact it tends to increase deterioration. The light phosphate coating tends to give protection and does provide marked protection under severe corroding conditions. The chromate treat-

TABLE II.—BEHAVIOUR OF ENAMELLED ZINC-BASE DIE-CASTINGS UNDER SALT SPRAY CONDITIONS.

Finish No.	Enamel Finish	Pre-treatment	Period of Salt Spray Test.		
			1 week	3 weeks	4 weeks
1	One coat cellulose enamel.	(a) None.	Slight corrosion from edges, adhesion rather poor.	Corrosion from edges, enamel adhesion poor.	Adhesion very poor.
		(b) Short period phosphate.	Unaffected.	Unaffected.	Slight corrosion and loss of adhesion near edges.
		(c) Long period phosphate.	Unaffected.	Slight corrosion at edges, enamel adhesion good.	Enamel adhesion still very good.
		(d) Chromate.	Unaffected.	Unaffected.	Unaffected.
		(e) Etch primer.	Exactly as (c).		
2	Two coats Cellulose enamel.	(a) None.	Adhesion poor.	Enamel adhesion very poor.	
		(b) Short period phosphate.			Enamel brittle but adhesion fair.
		(c) Long period phosphate.	Unaffected.	Corrosion from edges.	Enamel adhesion still very good.
		(d) Chromate.			Slight embrittlement of cellulose but enamel adhesion still very good. No corrosion.
		(e) Etch primer.	Exactly as (c).		
3	Two coats stoving enamel.	(a) None.	Adhesion poor.	Enamel finish blistered and peeling.	Corroded and condition very poor.
		(b) Short period phosphate.	Unaffected.	Unaffected.	Very slight corrosion at edges, and very slight embrittlement of enamel. Condition still good.
		(c) Long period phosphate.	Unaffected.	Slight corrosion from edges.	Except for slight corrosion at edges, enamel finish unimpaired, condition very good.
		(d) Chromate.	Unaffected.	Unaffected.	Unaffected.
		(e) Etch primer.	Exactly as (c).		
4	Stoved red oxide primer Stoved finishing enamel.	(a), (b), (c), (d).	Exactly as for finish No. 3.		
5	Stoved chromate primer. Stoved finishing enamel.	(a), (b), (c) and (d)	Exactly as for finish No. 3.		
6	One coat stoved aluminium.	(a) None.	Slight softening.	Slight softening.	Enamel rather soft and adhesion poor.
		(b) Short period phosphate.			Very slight corrosion at edges and very slight softening of enamel. Adhesion fairly good.
		(c) Long period phosphate.	Unaffected.	Slight corrosion from edges.	No further change, adhesion still very good.
		(d) Chromate.	Unaffected.	Unaffected.	Unaffected.
		(e) Etch primer.	Exactly as (c).		
7	One coat stoved aluminium. One coat stoved varnish.	(a), (b), (c), (d) and (e).	Exactly as for finish No. 6.		
8	One coat aluminium cellulose.	(a) None.	Slight softening.	Adhesion rather poor.	Adhesion very poor.
		(b) Short period phosphate.			Slight corrosion at edges, slight softening and slight loss of adhesion.
		(c) Long period phosphate.	Unaffected.	Slight corrosion from edges.	Enamel finish still very good.
		(d) Chromate.	Unaffected.	Unaffected.	Unaffected.
		(e) Etch primer.	Exactly as (c).		

TABLE III.—BEHAVIOUR OF ENAMELLED ZINC-BASE DIE-CASTINGS UNDER WARM HUMIDITY CONDITIONS.

Finish No.	Enamel Finish	Pre-treatment	Period of Humidity Tests.				
			1 month	2 months	6 months	1 year	2 years
1	One coat cellulose enamel.	(a) None.	Enamel brittle and easily flaked.				
		(b) Short period phosphate.		Enamel brittle.	Adhesion rather poor.		
		(c) Long period phosphate.					Slight embrittlement but finish still good.
		(d) Chromate.					ditto.
		(e) Etch primer.	Unaffected.	Unaffected.	Unaffected.	Slight embrittlement but finish still good.	
2	Two coats cellulose enamel.	(a), (b), (c) and (d).	Exactly as for finish No. 1.				
3	Two coats stoving enamel.	(a) None.	Enamel soft and easily peeled; finish very poor.				
		(b) Short period phosphate.	Unaffected.	Unaffected.	Slight embrittlement.	No further change.	Enamel slightly brittle brittle but adhesion still good.
		(c) Long period phosphate.	Unaffected.	Unaffected.	Unaffected.	Unaffected.	Entirely unaffected.
		(d) Chromate.	,,	,,	,,	,,	,, ,,
		(e) Etch primer.	,,	,,	,,	,,	
4	Stoved red oxide primer. Stoved finishing enamel.	(a), (b), (c), (d) and (e).	Exactly as for finish No. 3.				
5	Stoved chromate primer. Stoved finishing enamel.	(a), (b), (c), (d) and (e).	Exactly as for finish No. 3.				
6	One coat stoved aluminium.	(a) None.	Enamel soft and blistered.	Adhesion very poor.			
		(b) Short period phosphate.	Unaffected.	Unaffected.	Unaffected.	Unaffected.	Almost unaffected.
		(c) Long period phosphate.	,,	,,	,,	,,	Entirely unaffected.
		(d) Chromate.	,,	,,	,,	,,	,, ,,
		(e) Etch primer.	,,	,,	,,	,,	
7	One coat stoved aluminium. One coat stoved varnish.	(a), (b), (c), (d) and (e).	Exactly as for finish No. 6.				
8	One coat aluminium cellulose.	(a) None.	Little change.	Adhesion poor.	Adhesion poor.		
		(b) Short period phosphate.	Unaffected.	Unaffected.	Unaffected.	Unaffected.	Almost unaffected.
		(c) Long period phosphate.	,,	,,	,,	,,	Entirely unaffected.
		(d) Chromate.	,,	,,	,,	,,	,, ,,
		(e) Etch primer.	,,	,,	,,	,,	

ment gives real protection under all the tests imposed.

In the tabulated data showing performance of enamelled specimens under the various test conditions, specimens having the etch primer (applied to die-castings cleaned only by means of trichlorethylene vapour) are included. This primer is supplied in two parts, the resin base of poly-vinyl butyral in a solvent of mixed alcohols and water, and the phosphoric acid constituent in mixed alcoho's and water. The two parts are mixed in specified proportions before use and must be used within four hours. The acid has several functions, including the gelling of the resin in the applied film, interaction with the alcohols and etching the metal base. It was applied by spray, and stoved for 10 to 15 minutes at 250° F. Air drying for 16 hours before applying the enamel coat is also satisfactory.

The enamel media used included cellulose media, heavy bodied grades, thinned to a viscosity of about 35 seconds Ford Cup No. 4 at 25° C., and stoving enamels of the oil-modified alkyd type, thinned to 30 seconds viscosity for spraying. They were stoved 30 minutes at 250° F. The test results are mainly self explanatory.

Table II covers the severely corroding salt spray

TABLE IV.—BEHAVIOUR OF ENAMELLED ZINC-BASE DIE-CASTINGS UNDER OPEN ATMOSPHERIC TESTS.

Finish No.	Enamel Finish	Pre-treatment	Period of Open Atmospheric Test.		
			1 month	2 months	6 months
1	One coat of cellulose enamel.	(a) None.	Adhesion nil.		
		(b) Short period phosphate.	Unaffected.	Very slight embrittlement of enamel.	Enamel slightly brittle but adhesion still good.
		(c) Long period phosphate.	,,	,,	,,
		(d) Chromate.	,,	,,	,,
2	Two coats cellulose enamel.	(a), (b), (c) and (d).	Exactly as for finish No. 1.		
3	Two coats of stoving enamel.	(a) None.	Adhesion nil.		
		(b) Short period phosphate.	Unaffected.	Unaffected.	Unaffected.
		(c) Long period phosphate.	,,	,,	,,
		(d) Chromate.	,,	,,	,,
4	Stoved red oxide primer. Stoved finishing enamel.	(a), (b), (c) and (d).	Exactly as for finish No. 3.		
5	Stoved chromate primer. Stoved finishing enamel.	(a), (b), (c) and (d).	Exactly as for finish No. 3.		
6	One coat stoved aluminium.	(a) None.	Adhesion very poor.	Finish very soft and adhesion very poor.	
		(b) Short period phosphate.	Unaffected.	Unaffected.	Unaffected.
		(c) Long period phosphate.	,,	,,	,,
		(d) Chromate.	,,	,,	,,
7	One coat stoved aluminium. One coat stoved varnish.	(a), (b), (c) and (d).	Exactly as for finish No. 6.		
8	One coat aluminium cellulose enamel.	(a), (b), (c), and (d).	Exactly as for finish No. 6.		

TABLE V.—BEHAVIOUR OF ENAMELLED ZINC-BASE DIE-CASTINGS UNDER SEVERE HOT HUMIDITY CONDITIONS.

Finish No.	Enamel Finish	Behaviour under Hot Humidity Test.		
		1 day	3 days	20 days
9	Two coats cellulose enamel.	Finish embrittled, adhesion very poor.		
10	Short phosphate treatment and two coats of cellulose enamel.	Finish showing signs of crazing.	Finish cracked, adhesion poor.	
11	Etch-primer and one or two coats cellulose enamel.	Unaffected.	Unaffected.	Finish more brittle but adhesion still quite good.
12	Two coats stoving enamel.	Finish generally blistered.		
13	Short phosphate treatment and two coats stoving enamel.	Unaffected.	Finish generally blistered.	
14	Etch-primer and one or two coats stoving enamel.	Unaffected.	Unaffected.	Unaffected.

conditions. For every finish, the poor performance of enamel without pretreatment is apparent. The short phosphate treatment is seen to give marked benefit, and the long phosphate even better results. The etch primer at least equals the value imparted by the latter. The difficult chromate treatment is outstandingly the best.

Table III covers the mild humidity conditions and Table IV the open atmosphere tests. Exactly the same conclusions can be drawn.

Table V deals with the rather searching conditions of hot humidity; the outstanding superiority of the etch primer is very clearly brought out in these results.

Conclusions

In most cases, particularly those concerning large castings, zinc components can be handled more simply through chemical pre-treatment and enamelling processes than they can through electroplate finishing operations. Economics are in favour of enamelling; less trouble from chemicals retained in pores and recesses, and from blistering in service, are encountered. The "face" surfaces can be finished with the usual surfacings of filling and flatting to give really attractive blemish-free finishes. The above primarily demonstrates how adhesion and good durability can be assured. All the phosphate processes are effective, simple to apply and control, and economic in operation. The availability of good "wash" or "etch" primers further simplifies the problem, because aqueous chemical treatments can be eliminated, leaving the finishing as solely an enamelling shop proposition. These primers are a major advance in enamel finishing and their use promotes simplification as well as betterment of the finish. It is, therefore, considered that in a large number of cases nickel/chromium finishes can be replaced by enamel finishes with an improvement in serviceability and with little or no loss of decorative value.

Aero Research Summer School

The Technology of Synthetic Resin Adhesives

A DINNER at the Dorothy Café, Cambridge on September 28th, marked the end of the first Summer School devoted to synthetic resin adhesives and gluing techniques to be held in this country. The School had been held at St. Catherine's College, Cambridge, for the whole of the preceding week so that members could conveniently attend the series of morning lectures given in the lecture theatre of the University Department of Engineering. Several months before the opening of the School, all available places were taken, and St. Catharine's College was given the task of accommodating 150 visitors instead of the 50–80 originally anticipated. Most European nationalities were among the fourteen countries represented and two of the visitors from the British Commonwealth came from so far afield as Australia and New Zealand. Preliminary enquiries had shown that interests could be divided roughly into two main groups—those representing the woodworking industries, in which good gluing is a vital and integral part of manufacturing processes, and those representing engineering, electrical manufacturing and allied industries likely to need information upon adhesives of a different type. A division of the School into two courses was decided upon: a representative selection of woodworking concerns (Course I) would include members from plywood, furniture, aircraft, sports goods, and vehicle body building firms. In the wider range covered by Course II might be mentioned radio, rubber, brake linings, plastics, aircraft again and many fields of research, both industrial and governmental.

Morning lecture sessions were held daily, some lectures being given by members of the staff of Aero Research, Ltd., and others by specialists from different industries. Practical work and demonstrations were arranged for the afternoon at the Aero Research Works at Duxford, a village 10 miles south of Cambridge. There, again, the members of the two courses followed their specialised interests in using adhesives themselves and in seeing demonstrations of specal equipment.

Copies of all lectures given at the Summer School were distributed to all members and the book of the Course will be available within the next few weeks under the title "Structural Adhesives." This will be a fully-illustrated work and will cover the lectures given on both courses. The price is not expected to exceed 15s. 6d. and orders may be sent direct to Aero Research, Ltd., Duxford, Cambridge, or to the following booksellers: Books & Careers, Ltd., Oxford Street, London, W.1; B. H. Blackwell, Ltd., 48–51, Broad Street, Oxford; and W. Heffer & Sons, Ltd., 3 and 4, Petty Cury, Cambridge.

Blast-Furnace Relined in Record Time

ON Wednesday, September 26th, No. 10 blast furnace was blown-in at the Appleby-Frodingham Steel Co. (Branch of The United Steel Companies, Ltd.), after a reline which is believed to be the quickest ever carried out in this country. The blowing-in took place 32½ days after blast was taken off. During this time a complete reline was carried out, the hearth being increased in diameter from 22½ ft. to 25 ft. Auxiliary equipment was overhauled and extensive modifications were made to adjacent railroads.

Radioactive Materials in Industrial Metallurgy

By Sheila M. Holgate

Now that radioactive isotopes are more readily available, their use in laboratory and plant investigations is increasing steadily. In this article, which was among the prizewinners in the Birmingham Metallurgical Society's 1951 *Essay Competition, a brief general discussion of radioactivity is followed by more detailed consideration of its use in metallurgy. A useful list of references is appended.*

RADIOACTIVE materials have been known since 1896 and small quantities of the radioactive form of nearly every chemical element have been made available by means of the cyclotron since 1935. It was not until the end of World War II, however, that the development of the chain reacting atomic pile made possible a neutron bombardment to an intensity far exceeding that of the cyclotron, and changed the scale of radioactive isotope preparation from laboratory level to one approaching mass-production. The latest list of such isotopes[1] includes 75 artificially radioactive materials, as well as 12 types of fission product from long irradiated uranium and 20 naturally occurring radio-elements. These advances, affecting as they do the economic considerations of price, have tended to bring the use of these materials to the fore in many branches of scientific research. Their use in metallurgical fields, although it has not been so obvious, is, nevertheless, of some value, and the object of this article is to give an account of their present and potential applications, while stressing that the latter are not to be found exclusively in research work.

Their utilisation lies mainly in the following fields:

(*A*) In fundamental and *ad hoc* research, mainly as radio-tracers.

(*B*) Metallurgical or chemical analysis—most useful for routine work.

(*C*) Inspection and production control—other than routine analysis, e.g., thickness testing, gamma radiography.

(*D*) By the use of their characteristic properties.

(*E*) As a source of energy—nuclear power.

There are several considerations affecting the use of radioactive techniques in metallurgical and associated processes:

For the first three purposes outlined above, they are probably the most sensitive of all techniques available, provided the optimum conditions are obtained—a qualification which will be explained in more detail later. This excellent recommendation, however, is counter-balanced by the severe limitation of their use to cases where no other technique is readily available, or where such techniques as are available are less appropriate for the purpose. This is on account of the health hazard surrounding the use of active material. Where the health hazards can easily be minimised, the employment of radioactive techniques are often justifiable.

The optimum conditions for a particular use are governed by the chemical properties of the radioactive material, the type and strength of radiation and its half-life. If any particular process is to be studied by means of a labelled element, as in categories (*A*) and (*B*) above, the labelled element may have the same chemical properties as the material in which it will be traced. The type and strength of radiation should be that which will be most efficiently detected or measured compatible with the minimum health hazard. Finally, the half-life, which is defined as the time taken for a source of given strength to be reduced by a half (this value being assessed because of the exponential type of decay characteristic of all radioactivity), should be such as just to be sufficient for the process under consideration. An unduly long half-life gives rise to a greater health hazard, on the other hand, some elements which would be suitable in all other respects have such a short half-life (say two or three minutes or hours) that their practical value is nil. Naturally, the foregoing considerations represent the ideal, and often in practice it is necessary to effect a compromise. The use of the larger amounts which are probably necessary for the last two purposes outlined above (*D*) and (*E*) is at present restricted, not only by the terrific hazard involved, but also by the cost of the material.

It is not proposed to deal with the hazard question here. Suffice it to say that certain precautions must always be taken. These include the wearing of certain items of protective clothing and as little handling as possible of radioactive sources. The question is dealt with as thoroughly as possible on present-day knowledge by the various publications on the subject.[2, 3, 4, 5, 6]

Radiation and its Detection

All processes employing radioactive materials depend on the use of radiation in some way or other. Therefore, it is now proposed to go into the types and strength of radiation and its detection. Even if the qualitative or quantitative assessment of radiation is not required for the purpose of the experiment or process which the radio element is performing, it is essential from the health standpoint.

In trying to revert to a stable state, the excess energy which a radioactive element possesses is liberated by means of the phenomenon known as radiation. The three main types of radiation most frequently utilised are:—

(*a*) Alpha particles (α). These particles are nuclei of helium, consisting of two protons and two neutrons, and are only emitted from the heavier naturally occurring radioactive elements, e.g., polonium. They have a range of just under 4 cm. in air, and for this reason are less easily detected (as will be seen later) than

(*b*) Beta particles (β). These are fast moving electrons and may have a range of up to several feet in air. Usually β radiation is accompanied by

(*c*) Gamma radiation (γ). Gamma rays are rays in the true sense of the word (α and β being corpuscular radiations), in that they are penetrating electromagnetic vibrations of short wavelength (less than 0·1 Ångstrom unit). They are similar to X-rays.

Other types of decay will not be dealt with here as the three foregoing are those which will be shown to be of most practical value.

As well as a knowledge of the type of radiation involved, the strength of activity is the basis for all measurements. This is assessed in millicuries, where 1 millicurie is $3{\cdot}7 \times 10^7$ disintegrations per second. Thus,

1 Curie $= 3{\cdot}7 \times 10^{10}$ disintegration per second
1 millicurie (m.c.) $= 3{\cdot}7 \times 10^7$ " " "
1 microcurie (μc.) $= 3{\cdot}7 \times 10^4$ " " "

The disintegration event corresponds to a "count" on some of the instruments used for estimation of activity.

Rate of emission is directly proportional to the mass of radioactivity present. The mass of an element equivalent to a curie depends on the mass of the atoms, the half-life and on the extent to which the radioactive form is diluted with carrier or stable isotopes. Calculations of mass of material from strength of radioactivity are based on the following formulae.

$$\text{Atoms per Gram} = \frac{\text{Avogadro's Number } (6{\cdot}06 \times 10^{23})}{\text{Molecular Weight}}$$

$$\text{Transformation number } \lambda = \frac{\text{Disintegrations per Second per Gram}}{\text{Atoms per Gram}}$$

$$T = \text{Half-life} = 0{\cdot}693\ L$$

where $L = \text{Average or Mean Life} = \frac{1}{\lambda}$

From the health standpoint, the amount of radiation or "dose" is measured in röntgens, 1 röntgen being the amount of radiation of γ-or X-rays which will produce, in 1 cubic centimetre of air at NTP, the amount of ionisation necessary to separate 1 e.s.u. of charge. It is approximately the dose received in 1 hour at a distance of 1 metre from 1 gram of radium. For β radiation, the "röngten equivalent physical" in short, R.E.P. is used. This is that quantity of β radiation which gives the same amount of energy to 1 gram of air as does 1 röntgen of X-radiation. The energy of radiation is measured in M.E.V., i.e., million electron volts. This is the energy imparted to 1 electron falling through an electrostatic field of 1,000,000 volts.

Some idea of the relatively small masses of substances which must be dealt with in the employment of radioactive techniques may be obtained, when it is stated that for applications (*A*) and (*B*) the order of the final measurements may be a few microcuries, for application (*C*) 5 millicuries to 1 curie may be required, and for application (*D*) up to 100 millicuries may be necessary.

Methods of Detection

These include mainly

(i) Ionisation Chambers.
(ii) Radiation Monitors.
(iii) Photographic Techniques.

Again, it is not the present purpose to give an exhaustive account of measuring methods, but the above three types have been selected as being of fairly common usage, especially in applications such as will be described later. Other types of counting and detection mechanisms include scintillation methods, crystal counters, proportional counters, electroscopes, cloud chambers, and even micro-calorimeters which may use the heat given off by the substances.[7] Further details may be found in the literature.[8, 9, 10, 11, 12, 13]

(i) *Ionisation Chambers*

It is on the property of ionisation which the various radiations possess to some degree that many of the measuring methods are based, and one of the basic forms of detection is the ionisation chamber. In principle, it consists of two electrodes at a high potential difference in a volume of ionisable gas. In proportion to the intensity of the radiation which the gas receives, a leakage current flows between the electrodes, and can be measured either directly or after amplification by the usual means.

(ii) *Radiation Monitors*

A typical embodiment of the above (p. 12 of reference No. 12) consists of a radiation sensitive device connected to a mains-operated indicating unit which has a meter calibrated in "counts per second." For β and γ radiation, the sensitive probe unit is a thin walled glass Geiger-Muller tube.

The Geiger tube detects β and γ radiation by means of an electron avalanche process which is caused by the ionisation which occurs when such radiation enters the thin outer wall of the cylinder, which may be of thin metal or metal sputtered glass and is the cathode. A fine concentric wire, often of tungsten, is the anode and the tube is filled with 90% argon and 10% ethyl alcohol vapour at a pressure of 10 cm. mercury. A high voltage, applied to the central wire, is in the region of 1,000 volts for the plateau region.

For detection of alpha particles, there is provision for the fitting of a probe which consists of a scintillation counter arranged to receive and register the small light flashes which occur when alpha particles enter a thin window and hit a scintillation screen. There is also provision for a thin window counter and a scaling unit for large scale counting. The instrument can be used for both qualitative and quantitative detection of radiation.

(iii) *Photographic Techniques*

These include some of the most generally useful detection techniques in existence, especially from the metallurgical point of view, where such techniques as autoradiography may be combined with metallography. Again, by means of their ionising properties, the radiations are capable of affecting a photographic emulsion. The effect of exposure of a photographic emulsion to a radioactive source is cumulative and on development the degrees of intensity of blackening give a measure of the radiation to which the emulsion has been subject. The effect varies with the specific ionisation formed and is therefore relatively sensitive to α, less to β and still less to γ. This form of detection can be used in a qualitative manner to locate radiation, or, when suitable calibrations are made, it can be used for quantitative measurement. More recently, special emulsions have been produced specially for this type of work. They are known as nuclear track emulsions and they facilitate

the microscopical counting of tracks made, each of which represent an atomic collision of some kind.

In all types of detection and counting, absolute measurements are not always essential, nor even desirable. Comparative measurements are often of much more practical importance as will be seen later. There are, however, certain factors which influence all types of detection and for accuracy of results, these should be taken into account.

For consecutive measurements in any experimental procedure, the geometry of the arrangement of detecting mechanism and sample being estimated should be accurately reproducible: this is especially important with Geiger-Muller counting arrangements. By careful consideration of this aspect, errors are reduced. The probable errors encountered in this type of counting may be summarised by the following :[9]

(*a*) Tube resolving time.
(*b*) Background correction.
(*c*) Counter efficiency.
(*d*) Absorption and scattering.
(*e*) Statistics.
(*f*) Decay scheme.

Factors (*a*) and (*c*) are governed by the apparatus use, while (*d*) and (*f*) are characteristic of the radioactive sample. Factor (*b*) can only be minimised by the taking of due precautions—e.g., use of lead-encased apparatus to prevent the stray radiations omnipresent and known as background. Since radioactive decay is a random process, the accuracy of measurement is limited by the statistical variations inevitably associated therewith. Some degree of mathematical analysis lessens the error relative to these variations. (Poisson's Law—Reference No. 10).

If the sources of probable error listed above, with the exception of those relating only to the Geiger tube, are taken into account when employing any method of measurement, a high degree of accuracy may be obtained.

Summary of Metallurgical and Associated Applications

It has been necessary up to this point to deal with the elementary principles underlying the use of radioactive materials in industry and research, in order that the examples of application might be more easily illustrated.

(*A*) Fundamental and *Ad Hoc* Research

Diffusion. Probably the best known researches in which radioactive tracers have been used are those conducted on diffusion measurements. The advantage of these materials is shown in that self-diffusion can be assessed by plating on to the specimen metal the radioactive element, and, after heating cycles at various temperatures, diffusion can be assessed by comparative counts obtained on a solution of thin layers of metal successively machined from the sample.

Such work has been carried out by many workers and diffusion rates for copper, silver, zinc, lead, gold, etc., have been established [12, 14].

Alloy Systems. In order to assist metallographic procedures, it has been found possible to investigate the distribution of elements in alloy systems; an example of this is the distribution of lead in other metals.[15] An autoradiographic method was used to locate the lead which only slightly soluble. The base metal was melted with the lead isotope in an atmosphere of hydrogen and allowed to crystallise. Uniform distribution, showing solubility of the lead in the parent metal gave a uniform blackening of the photographic plate, these metals in which it is practically insoluble, Bi, Sn, Sb, Ag, Au, Cu, Ni, showed the blackening at grain boundaries of the parent grains. A similar application was made by Stephens and Lewis in investigating the distribution of aluminium in silicon.[16]

Metal Production. In processes of metal production, radioactive tracing technique has perhaps filled a long felt need in providing a feasible method of investigating some problems.

Gas transit times in a driving blast furnace have been investigated by Voice.[17] In this application, a radioactive inert gas—radon—was used and the useful property of radioactivity being absolutely unaffected by extremes of heat or cold is seen to advantage. Eight millicuries of radon were projected by explosives into the hot blast stream through a tuyère, and samples of gas were taken at known intervals of time from a point on the stockline and were analysed for radioactivity. The gas transit time was then deduced from a time/activity curve. Initial experiments indicated much lower velocities than had been given by previous estimates.[18] Other similar investigations have been conducted on the flow pattern of hot gases by Mayorcas and Thring.[19] Such results are of value in furnace construction.

The study of the distribution of calcium between molten slags and iron saturated with carbon has also been carried out[22, 23] using isotopes. Radioactive calcium was used to detect the presence of calcium in various blast-furnace slags: no calcium was found in the metals which were produced under conditions which were favourable for the reduction of calcium. Conclusions were drawn, therefore, that the calcium content of the iron was, in practice, less than $6 \times 10^{-5}\%$.

In a further investigation, equilibrium conditions in the dephosphorisation of steel have been found to be established after 15 minutes in experiments using activated tricalcium phosphate.[24]

The source of sulphur in coke has been investigated to determine whether there was any advantage in making coke from coal containing pyritic or organic sulphur.[25] Several pounds of radioactive iron pyrites were added to 12 tons of coal prior to coking. During coking, checks on the total sulphur content of the gas and on the radioactive sulphur content of the gas were made, and after coking, the coke was analysed for total and radioactive sulphur content. It was found that the ratio of radioactive sulphur and therefore of pyritic sulphur, to the total sulphur content in the coke-oven gas was the same as the ratio in the original coal. The ratio was the same for the coke residue. There was, therefore, no point in selecting either type of coal in preference to the other.

It is possible to determine the relative volatility of various components of alloys in their production. For example, the rate of volatilisation of zinc in the making of brass has been measured,[20] and although no details of the technique are given, an isotope of zinc is, presumably, added to the melt and comparative measurements taken of samples of the metal during melting.

In the field of electrodeposition, experiments have been carried out to determine the valency state of the metal which is deposited[21]. By tagging the trivalent or hexavalent chromium in a chromic acid plating bath with a radioactive isotope of chromium, these workers found on examination of the deposit for radioactivity that the metal is deposited directly from the hexavalent

chromium present in the bath. The tracer used was chromium 51, a soft gamma emitter with a half life of 26·5 days. The deposit was plated on a brass cylinder within which a thin walled glass gamma ray counter was placed.

Surface Processes. The tracer technique affords a useful means of investigation of surface processes such as corrosion, catalytic activity, friction studies, metal transfer and the physics of lubrication.

References to the use of tracers in corrosion studies are given by Williams and Finniston[14] and Taylor[12]. Work has been carried out[25] in which radioactive iron—Fe 55 and Fe 59—was plated on to iron specimens subject to corrosive conditions and auto-radiographed. These were compared with uncorroded specimens as controls. Removal of iron and oxide formation results in a decrease in density of the blackening.

Surface phenomena on solids have recently been studied by Frauenfelder.[26] He has developed new methods for measuring desorption probabilities, sticking coefficients and surface diffusion coefficients using radioactive substances.

Friction studies and metal transfer problems lend themselves well to solution by means of radioactive tracer technique. The softer of two surfaces in contact is activated and the traces of this metal transferred to the harder metal can be detected by auto-radiography.[27] Some of the available methods of activating the surface under test are : irradiation of the surface in a cyclotron with a stream of deuterons ; electroplating a thin layer of metal containing an isotope on to the surface and subsequent heating for uniform diffusion ; adding an isotope to the metal during melting ; or, irradiating the whole material by slow neutrons in an atomic pile. The first two methods keep the activity near the surface, the other two distribute it uniformly throughout the specimen. Friction tracks are produced by the softer metal on the harder surface and show on the auto-radiograph. In one case of mild steel running on alloy steel, the transferred metal is shown to adhere in discrete spots, possibly corresponding to local welds. The same author makes a reference to the use of oil bearing a trace of radioactive sulphur for investigating lubrication techniques. According to some workers, effective lubrication is only obtained when chemical reaction occurs between adsorbed lubricant films and metal surfaces.[28] Metal transfer from piston rings to cylinders during running, using radioactive chromium and an auto-radiographic technique, and metal transfer in cutting processes have been investigated by similar methods.[29, 30]

Mineral Dressing. Reference is made[28] to the use of sulphur 35 and carbon 14 in research on chemical activation in flotation processes and on ores by neutron activation for picker-belt separation. This research was carried out by the department of metallurgy of the Massachusetts Institute of Technology under the auspices of the Atomic Energy Commission.

(*B*) Metallurgical and Chemical Analysis

It can easily be deduced, from what has been said in (*A*), that there is a wide scope for the use of tracers in analysis to supplant, in some cases, the more conventional methods. Quite often, the two fields of use actually overlap. The following examples are quoted as being of interest.

The addition of radioactive phosphorus to the melt in steel production enables an estimation of the final phosphorus content in the slag and steel and check estimations during the process by Geiger counting methods.[15] Trace substances in metals, difficult to detect by normal chemical analysis means, may be assessed by means of an activation technique.[20] Sulphur and selenium are typical of such impurities. Ten parts per million of selenium and 50 parts per million of sulphur can be estimated in the following manner. A known weight of the metal or alloy is irradiated in an atomic pile for a known time, dissolved and a small amount of stable carrier sulphur and carrier selenium added. After chemical separation of the sulphur and selenium the final specific activity is examined by means of a Geiger counter, the result depending on the amounts of sulphur and selenium initially irradiated.

Such processes as described, show how useful is the property whereby the radioactivity is distributed proportionally in aliquot portions of the whole.

(*C*) Inspection and Production Control

Into this category fall the techniques of thickness measurement and detection of surface and internal defects. The former has been effected with suitable beta and gamma emitters and the latter embraces the field of gamma-ray radiography. For a full consideration of this subject a voluminous review could be devoted to it entirely. It is not possible to deal with it adequately in the space available, but an attempt will be made to cover the salient features. It is sometimes forgotten, so vast is this field, that in reality it is only a modified form of thickness measurement.

Thickness measurement is based on the fact that part of the radiations from a radioactive source are absorbed by material interposed between them and a counting device. One method of employment of this technique is to have parallel systems whereby there are two sources, the radiations from one of which are counted after passing through a known standard thickness and the radiations from the other are counted after passing through the unknown thickness. With suitable calibration, the measuring device can be made to read directly in terms of thickness, provided the material is all of the same density. Provided recalibration is made to allow for the decay of the source this sytem is adequate for single measurements, but for continuous control the former system is the better. For thicknesses over those of a few thousandths of an inch, gamma-ray emitters or X-ray emitters are more suitable. Accuracy of the method is claimed to be $\pm$ 0·05% of sample thickness, or a mass of $\pm$ 0·03 mg./sq. cm., whichever is greater.[25] Strontium 90, rubidium 106, cobalt 60, cerium 137, and sulphur 35 are suitable beta emitters which also have some gamma radiation. Recently, a complete patent specification has been published giving an improved method for thickness measurement, said to be suitable for steel strip.[31] Actually this invention employs an X-ray tube and an ionisation chamber receives the radiations through the steel strip, but the principle is the same as that for a beta or gamma isotope source. Various electrical improvements in the response of the apparatus to the radiations enable adjustments to the rolls to be made without undue loss of time when the strip is coming through at speed.

X-ray radiography is a well-known type of non-destructive testing and has proved of great value for many years. Now that there are available many gamma ray emitters,[32] fairly cheap to purchase, testing which would otherwise not be carried out, due to the cost of installing X-ray equipment, is now possible. The

process of radiographing with radioactive isotopes instead of X-ray tubes differs from X-ray work in slight modifications of technique, exposure, resolution obtained, etc. The supreme advantages which the use of such sources confers is that of low cost and (provided the proper precautions are taken) transportability. That is to say, pieces of equipment far too big to be tested by X-ray laboratories can be radiographed where they have been constructed. An example of this appeared recently in the press which described the testing of the welds of the biggest pressure gas-holder ever to be constructed in Britain, and probably in the world.[33]

For protection during storage and transport, the sources used may be housed in special containers which prevent the escape of radiation. The source may either be left in this container, which has a diaphragm which allows the rays to pass in one direction, or may be removed entirely. In this instance, since the unshrouded source emits full intensity in all directions, multiple exposures may be made by arranging the specimens all round the source.[34, 35] Herein lies another of the advantages of gamma radiography, and since the exposure is longer than that for X-ray work, the facts that such an arrangement can be left overnight and that multiple exposures can be made, offset this disadvantage. In the same way, hollow cylindrical vessels may be tested by placing the source somewhere inside the vessel and placing the film all round the outside. This may be done on vessels up to about 3 ft. in diameter. Radium, radon, tantalum 182 and cobalt 60 are those sources most frequently used and their respective performances have been compared.[36] Details of technique cannot be adequately dealt with, but much of interest is to be found in the literature on the subject.[1, 32, 34, 35, 36, 37, 38]

Surface defects such as cracks are reported to be capable of detection by applying a greasy paste containing radioactive material to the surface of the metal under high pressure. Upon cleaning the surface, the radioactive paste is left in the cracks and can be detected by auto-radiographic means.[14, 15]

(*D*) By the Use of Characteristic Properties

One of the most useful characteristics of the radiations is their property of ionisation. Alpha emitters are the best ionisers and to a less degree beta emitters have the property.

The dispersal of static electricity from moving belts and machinery has been effected by the use of alpha emitters and this process makes for better working conditions than result from the maintenance of high humidity.[20, 25] Unfortunately, polonium which, is one of the best alpha emitters has certain strong disadvantages from the health standpoint.[3] Further, the best ionisers, alphas, only have a short range in air—about 4 cm.—and so the source has to be placed close to the area to be treated.

Similar use is made of radioactive materials in electrical discharge devices such as gas discharge tubes and sparking plugs on automobile engines.[25, 39, 40] It is claimed for the first use that the striking voltage variation can be reduced from 50% to 10%, and for the second that the engine may be started in the cold more easily.

More truly a metallurgical application, is the use of radioactive isotopes and natural radioactive material in the control of the arc in electrical metal spraying pistols. It is claimed that the ionisation of the gaseous medium in the arc gap helps to prevent wander and rupture of the arc.[5]

(*E*) As a Source of Energy—Nuclear Power

It is perhaps to this aspect of atomic energy that the industrialists are looking with most hope. As yet, the whole project is in the speculative stage. Perhaps it is not outside the bounds of possibility that the metallurgists of the future will be able to design furnace installations, the source of heat for which is derived from atomic fuel. The obstacles yet to be overcome of course are numerous. Such an installation would have to be adequately screened so that it would be safe to approach; new metals and alloys would have to be developed capable of withstanding the high temperatures encountered within the atomic reactor; and cooling systems for temperature control would have to be devised. Moreover, much more research is needed on the effect of prolonged neutron bombardment on metals and alloys. Work on nuclear reactors for power production is being carried on in the various atomic research establishments in the world, and even now it is speculated that nuclear furnaces based on present research may be able to be operated without refuelling for between 10 and 30 years.[42] Sparse though the literature on this subject is, it serves to show that progress will be possible, but not, perhaps, for the next 25 years will any full-scale applications be made.[43, 44, 45]

Conclusion

An attempt has been made to present the present-day metallurgical applications of radioactive materials and their associated techniques. From these examples, further applications are easily seen to be possible* and many more research problems may be attacked in this way. The plastic flow of metals under extrusion could be investigated with a modification of the tracer technique; pick-up of die metal in tube and wire-drawing operations could be assessed; and, altogether, many works problems could be tackled.

It has been the aim throughout to exclude all but the details of practical interest. The complicated physical and mathematical aspects can be more fully perused by reference to the literature. It would not be possible to adopt the methods of investigation outlined without further study of the subject, and here it must be stressed once more that such methods should not be used unless they hold distinct advantages over any others available and, unless a specialist on the subject, without a full consultation of the personnel of the advisory service of the Atomic Energy Research Establishment, Harwell. This service provides for excellent guidance in the choice of suitable materials and methods and is almost unique in giving help and guidance to the scientist and industrialist alike in the utilisation of atomic energy for the progress and welfare of industrial civilisation.

Acknowledgment

The author wishes to thank the Birmingham Metallurgical Society for permission to publish this paper.

* This paper was written at the end of 1950 and certain of the potential applications listed here have now actually been used in practice.

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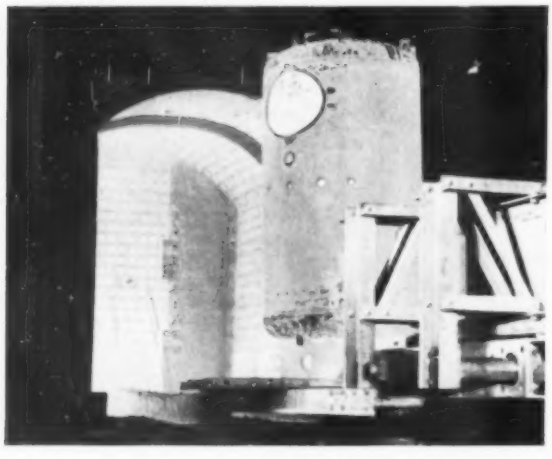
New Works Cinema

ONE of the first industrial concerns in the country to realise the immense possibilities of the industrial film, The Carborundum Company, commenced serious use of the medium around 1920. Since that time films have played a big part in publicizing the firm's products, and in instructing operatives and others in their correct use. To-day, all the films are made in colour and are the work of the company's own film production unit. The two most recent releases were shown on October 2nd on the occasion of the opening of the Company's new cinema at the Trafford Park Works.

The first of the films shown illustrated a highly specialised application of Carborundum abrasives. Made in association with Sir Howard Grubb, Parsons & Company, and by kind permission of the Astronomer Royal, "The Grinding of a 98 in. Telescope Mirror," entailed a number of visits by the Film Unit to Newcastle over a period of some eighteen months.



Courtesy of Enamelled Metal Products, Ltd.

A shot from "The Super Refractories" shows a large muffle furnace by Carborundum used for the glass enamelling of tanks. The effect of the high conductivity of Carbofrax is strikingly shown in this photograph by the dark section in the corner of the muffle where the wall is built of ordinary firebrick.

The new cinema.

In contrast the second film, "The Super Refractories," was made in a number of important plants, including the Royal Mint. The film opens with a visit to the new factory at Rainford which has been opened solely for the production of the Carborundum range of refractories. Subsequent sections show the use of these refractories in furnace construction, as skids in the hot zones of billet heating furnaces, as crucibles for metal melting, and as kiln furniture in the pottery industry.

The cinema is capable of seating comfortably some 200 people and is equipped with modern projection and sound apparatus. The wall-panels and baffles suspended from the roof serve the dual purpose of decoration and sound absorption, since the acoustics of the original building were not too good. The projection room itself is also employed for film editing, sound dubbing of new film, and for the making of the animation scenes which figure in many Carborundum productions. The theatre will be used for many purposes, such as for the entertainment of overseas visitors, the instruction by film of apprentices, visiting engineering societies and the like, and for the Company's own internal conferences. It will also serve as a planning and try-out centre for the exhibitions in which the firm regularly participates.

Weathering Tests of Tin-Zinc Alloy Coatings on Steel

By S. C. Britton and R. M. Angles
Tin Research Institute

The information which has already been published on the behaviour of electro-deposited tin-zinc alloy coatings in salt-spray and humidity tests is supplemented by the results of weathering tests in urban, suburban and marine atmospheres. In urban and suburban atmospheres, the protection afforded by coatings of equal thickness diminished in the order—zinc, 50/50 tin-zinc alloy, 80/20 tin-zinc alloy, cadmium. By the sea, an alloy with 50% tin was rather better than zinc and cadmium, but an 80% tin alloy was inferior to zinc and no better than cadmium. In tests involving more continuously humid conditions, tin-zinc alloy was superior to zinc.

ALTHOUGH the use of electro-deposited coatings of tin-zinc alloy is now well established commercially, the behaviour of the coatings when exposed to the weather has not previously been described.

Accounts have been given of the deposition process[1,2] and of comparisons of the deposits with other protective coatings in salt-spray and humidity tests[3,4]. The accelerated corrosion tests showed that coatings containing 50–80% tin, of thickness greater than 0·0003 in., gave good protection against rusting at pores and were themselves much less disfigured by corrosion-product than zinc.

Outdoor exposure tests of coatings of tin-zinc, cadmium, tin and zinc on steel were started in 1947 at three places. Sufficient progress has now been made to permit an assessment of the comparative merits of these coatings.

Materials

The steel sheets on which the coatings were deposited were all drawn at random from the same supply of cold-rolled, close-annealed auto-body stock 0·036 in. thick. The steel contained: C, 0·15%; Mn, 0·35%; Si, 0·010%; P, 0·065%; S, 0·083%; Cu, 0·13%; Cr, 0·038%; Ni, 0·16%. All test panels were of size 12 × 8 in.

Zinc, cadmium, and tin-zinc alloy were deposited from conventional cyanide baths and tin from a sodium stannate bath. Two compositions of tin-zinc alloy, 80% and 50% tin, were employed. Each metal was tested in thicknesses of 0·0003 in., 0·0005 in., and 0·001 in.

Two panels were exposed for every coating except tin at all test-locations, but one of each pair received before exposure an appropriate "passivation" treatment. The treatments used were:

Zinc and Cadmium.—Immersion at room temperature in a solution of sodium dichromate, acidified with sulphuric acid, as laid down in Ministry of Supply Specification D.T.D. 923 of January, 1945.

Tin-Zinc.—Immersion for 30 seconds in 2% chromic acid at 80° C.

Before exposure, each panel received a single scratch, 5 in. long, perpendicular to the bottom edge of the panel at its central point. This scratch, made by hand with a rat-tail file ground to a point, penetrated the coatings.

Methods

Three exposure sites were used:

1. *Perivale*, on the roof of the Tin Research Institute, well above surrounding buildings, providing exposure to a suburban atmosphere, polluted mainly by domestic coal smoke, with an annual rainfall of about 25 in.
2. *Oldham*, on the unsheltered, flat roof of a factory building, providing exposure to an atmosphere receiving a good deal of industrial smoke, with an annual rainfall of about 35 in.
3. *Hurst Castle*, close to the edge of the sea, and about 50 feet above it, on the south coast of England, providing an atmosphere of a purely marine type, free from smoke, but with occasional wind-driven light sea-spray. The annual rainfall is about 30 in.

The panels were all held at an angle of 45° to the horizontal. At Perivale and at Oldham, they were secured by light contact of the side and bottom edges with porcelain bobbin insulators of 1·5 in. diameter. The insulators were attached by screws to 2 in. wooden battens which were clear of the backs of the panels by about ½ in. At Hurst Castle about ¼ in. at the top and bottom of the panel was gripped between wooden battens.

Inspections were normally made at 3-monthly intervals. The time for the first appearance of rust and, later, the percentage of area covered by rust were recorded.

Sets of panels, withdrawn from the tests when many of them were completely rusty, were subjected to cupping tests, using the Avery Sheet Metal Tester. This device is similar in principle to the Erichsen Machine. A hardened steel ball of 2 cm. diameter is pressed into the sheet under test and the depth of cup at fracture of the sheet is measured. It was hoped that such a test would give a quantitative estimate of the deterioration of the panels in a way which would reveal any localised corrosion. The use of a cupping test was shown to be potentially valuable for this purpose by Portevin[5].

Results

I.—Visual Observations

Coatings other than Tin.—The breakdown of all the coatings except tin followed a similar trend. There was a period of freedom from rust, except within the scratch, during which slight white corrosion product appeared on the coatings. Zinc coatings darkened at Perivale and Oldham; the others became dull but retained their colour. Rust usually first appeared round the scratches and on the edges and gradually spread until the whole surface was covered.

In the marine exposure at Hurst Castle, the develop-

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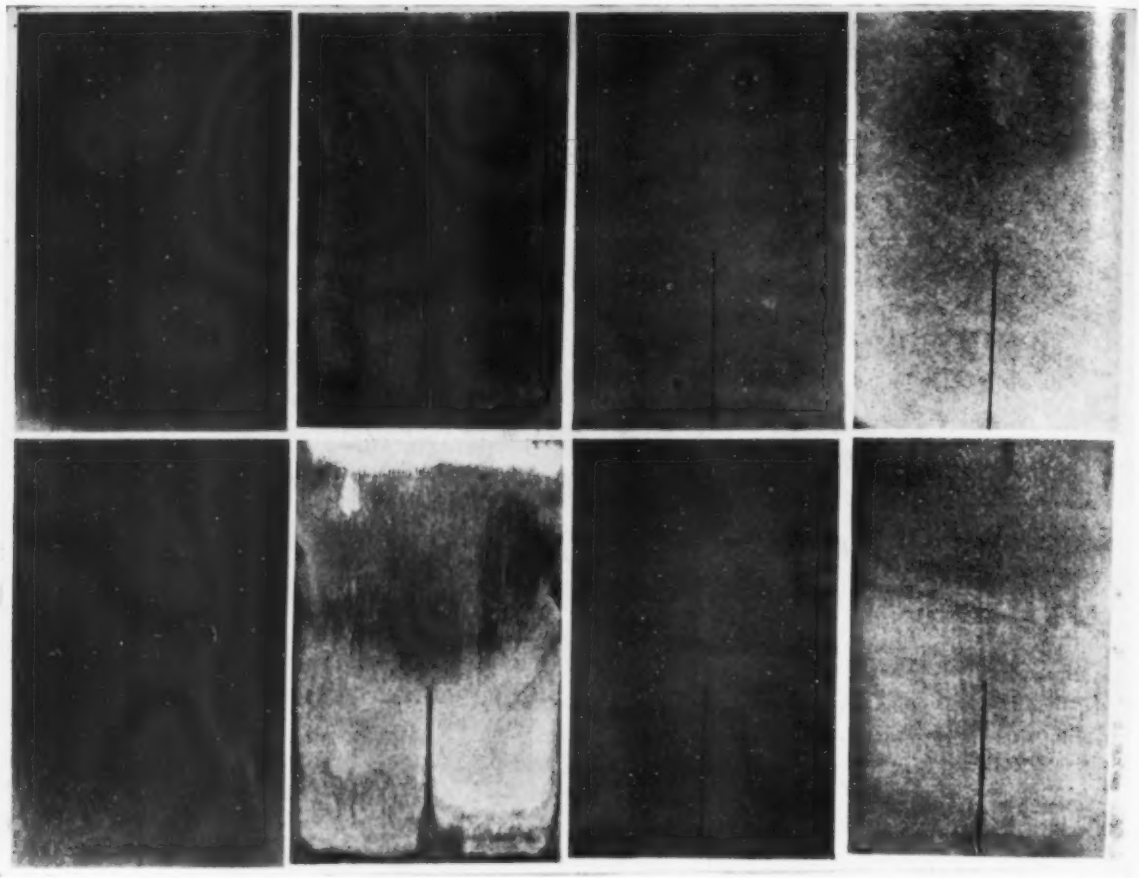

Fig. 1.—Panels with 0·0005-in. coatings after 18 months exposure at Perivale (suburban). Those on the lower row were "passivated." The coatings are (left to right): Cadmium, 80/20 Tin-Zinc, 50/50 Tin-Zinc, Zinc.

ment of continuous rust was preceded by the formation of discrete rust spots which gradually merged. Elsewhere the initial stage of rusting was the formation of a continuous patch of brown stain which gradually changed into rough red rust. Nowhere was there any visible sign of pitting of the steel.

The scratches on all samples showed traces of rust at an early stage, but this rapidly became converted to a hard dark product and further action within the scratch appeared to stop. The usual initiation of general rusting round the scratches points to some sacrificial protection of the scratches by all the coatings.

Photographs of panels from Perivale and Hurst Castle taken at stages which indicate the relative order of merit are reproduced in Figs. 1 and 2. Panels from Oldham carried too much dirt for satisfactory photographic reproduction in black and white, but the relative rate of deterioration of the coatings is fairly well represented by the Perivale panels.

The under-sides of the panels, sheltered from rain, deteriorated much more slowly than the upper surfaces. The relative performance of the various coatings in delaying the appearance and the spread of rust was similar for both sides, although the superiority of tin-zinc over cadmium was more marked on the under-side. The condition of the under-sides of a set of panels exposed at Perivale is illustrated by Fig. 3. Before rusting, the tin-zinc alloy coatings, especially those with 80% tin, remained lighter, brighter and less disfigured by white corrosion product than the zinc coatings. The illustration gives a faint indication of this.

Tin Coatings.—Tin had a distinctive behaviour everywhere. Rust spots became visible immediately, except on the 0·001 in. coating at Oldham, but at Perivale and Oldham these disappeared from the upper faces of the panels after a few months and the panels became covered with a thin white-spotted grey layer of corrosion product of the coating. After a further period, depending on the coating thickness, some brown stain appeared in the layer of corrosion product. Rust spots formed on the under-sides of the panels in the early stages remained visible but increased only very slowly. At Oldham after four years, the panel coated with 0·001 in. of tin had a better general appearance on both sides than any other panel there.

At Hurst Castle, the rust spots on both sides of the tin-coated panels increased steadily in size and number and the coating also suffered conspicuous corrosion. Presumably, here, the greater conductivity of the water in the pores of the coating permitted some galvanic action between coating and steel, whereas, elsewhere, the resistance to current flow was too high for any stimulation of rusting at the pores which became blanketed by corrosion product of the coating. The scratches on tin-coated panels rusted very slowly at Oldham and Perivale, and

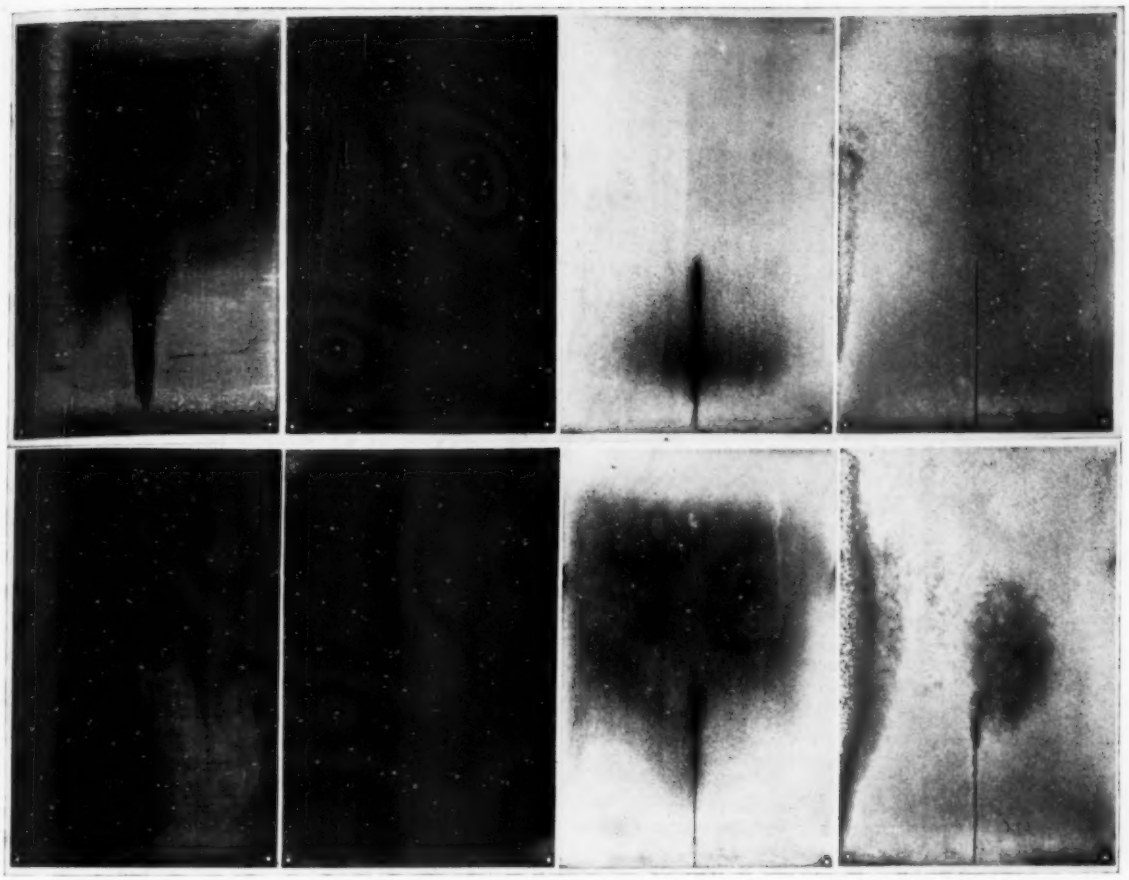

Fig. 2.—Panels with 0·0005-in. coatings after 44 months exposure at Hurst Castle (sea coast). Those on the lower row were "passivated." The coatings are (left to right): Cadmium, 80/20 Tin-Zinc, 50/50 Tin-Zinc, Zinc.

more rapidly at Hurst Castle, throughout the exposure.

The times elapsing for the first appearance of rust on the various coatings and for the whole upper sides of the panels to become rusted are shown in Tables I–III. Complete rust cover of a panel was taken as the stage at which the whole surface was first seen to be actively rusting. The whole surface of some panels was rust-stained before this, but as this was sometimes due to the

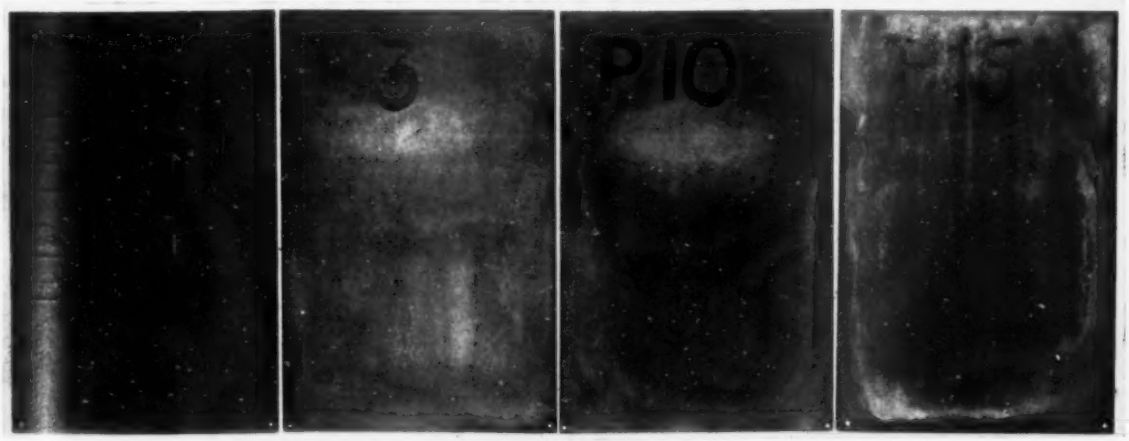

Fig 3.—Under-sides of panels with 0·0005-in. coatings after 44 months exposure at Perivale. The coatings are (left to right): Cadmium, 80/20 Tin-Zinc, 50/50 Tin-Zinc, Zinc.

TABLE I.—MONTHS OF EXPOSURE AT OLDHAM FOR FIRST APPEARANCE OF RUST (A) AND FOR PANEL TO BECOME COMPLETELY RUST COVERED (B). PANELS WITHDRAWN BEFORE BECOMING COMPLETELY RUSTY HAVE PERCENTAGES OF AREA RUSTING SHOWN.

Coating Metal	Coating Thickness 0·0003 in.		0·0005 in.		0·001 in.	
	A	B	A	B	A	B
Zinc	11	90% at 18	24	80% at 44	29	20% at 44
Zinc (passivated)	18	70% at 18	24	80% at 44	32	10% at 44
Tin-Zinc Alloy (50% Tin)	4	11	11	25% at 18	11	24
Tin-Zinc Alloy (50% Tin) (passivated)	4	18	7	40% at 18	11	35
Tin-Zinc Alloy (80% Tin)	4	18	4	18	7	30% at 18
Tin-Zinc Alloy (80% Tin) (passivated)	4	25% at 18	4	50% at 18	7	10% at 18
Cadmium	2	7	4	11	7	18
Cadmium (passivated)	4	7	4	7	7	18
Tin	1	0% at 18	1	0% at 44	24	0% at 44

TABLE II.—MONTHS OF EXPOSURE AT PERIVALE FOR FIRST APPEARANCE OF RUST (A) AND FOR PANEL TO BECOME COMPLETELY RUSTY (B). PANELS WITHDRAWN BEFORE BECOMING COMPLETELY RUSTY HAVE PERCENTAGE OF AREA RUSTING SHOWN.

Coating Metal	Coating Thickness 0·0003 in.		0·0005 in.		0·001 in.	
	A	B	A	B	A	B
Zinc	20 more than	10% at 26	37	10% at 44	more than 48	0% at 48
Zinc (passivated)	26	0% at 26	37	5% at 44	48 more than	0% at 48
Tin-Zinc Alloy (50% Tin)	14	25	19	37	31	80% at 48
Tin-Zinc Alloy (50% Tin) (passivated)	14	25	19	37	31	70% at 48
Tin-Zinc Alloy (80% Tin)	7	14	14	28	14	31
Tin-Zinc Alloy (80% Tin) (passivated)	8	14	14	32	14	37
Cadmium	6	9	8	19	14	31
Cadmium (passivated)	8	11	8	19	14	25
Tin	2	26	3	0% at 44	3	0% at 48

TABLE III.—MONTHS OF EXPOSURE AT HURST CASTLE FOR FIRST APPEARANCE OF RUST (A) AND FOR PANEL TO BECOME COMPLETELY RUSTY (B). PANELS WITHDRAWN BEFORE BECOMING COMPLETELY RUSTY HAVE PERCENTAGE OF AREA RUSTING SHOWN.

Coating Metal	Coating Thickness 0·0003 in.		0·0005 in.		0·001 in.	
	A	B	A	B	A	B
Zinc	18	20% at 29	33	10% at 45	36	5% at 48
Zinc (passivated)	18	10% at 29	18	20% at 45	36	5% at 48
Tin-Zinc Alloy (50% Tin)	25	10% at 29	35	5% at 45	more than 48	0% at 48
Tin-Zinc Alloy (50% Tin) (passivated)	29	5% at 29	35	20% at 45	more than 48	0% at 48
Tin-Zinc Alloy (80% Tin)	9	21	18	38	36	45% at 48
Tin-Zinc Alloy (80% Tin) (passivated)	13	29	21	40	36	25% at 48
Cadmium	8	21	21	70% at 45	34	10% at 48
Cadmium (passivated)	13	25	21	80% at 45	25	40% at 48
Tin	1	12	1	60% at 18	1	50% at 18

flow of rust from an actively rusting area over the remainder of the surface, the general formation of loose rust was the easiest to define and observe.

In general, the comparative merits of the various coatings are the same for every thickness and also for "passivated" and "as plated" coatings. At Hurst Castle, the 80/20 tin-zinc coatings were slightly superior to cadmium coatings for a thickness of 0·0003 in. and slightly inferior at the greater thicknesses, but otherwise the indications which emerge are clear.

1. *At Oldham and Perivale*, the zinc coating was superior to any other in preventing the appearance of rust and in delaying complete rusting. The tin-zinc alloy coatings were better than cadmium in delaying complete rusting, and were in no instance worse in preventing the first appearance of rust. The spread of rust in general was appreciably more rapid with the cadmium coatings than with the alloy coatings. Tin was much superior to the alloy coatings in preventing serious rusting, but permitted the early appearance of rust.

2. *At Hurst Castle*, the results were rather different. Here the 50% tin-zinc alloy was the most protective coating, with zinc as next best, although cadmium and the 80% tin-zinc alloy were little inferior to zinc for 0·001 in. coatings. The rate of rust-spread on cadmium was here no greater than on the other coatings and the performances of cadmium and the 80% tin-zinc alloy are, over-all, about equal. Tin coatings were decidedly inferior to the others.

3. The effect of the passivation treatments was in no instance large. Considering the time to first appearance of rust, the qualitative effect was :

	Better	*No Effect*	*Worse*
Zinc	3	4	1
50% Tin-Zinc Alloy	1	6	1
80% Tin-Zinc Alloy	3	6	0
Cadmium	3	5	1

TABLE IV.—CUP-DEPTHS (MM.) TO FRACTURE IN AVERY SHEET METAL TESTER OF SHEETS EXPOSED AT OLDHAM. EACH VALUE IS DERIVED FROM 12 TESTS.

Coating.	0·0003 in. coatings exposed for 18 months.		0·0005 in. coatings, tin and zinc exposed for 44 months, others for 18 months.	
	Mean	Standard Deviation	Mean	Standard Deviation
Zinc	7·80	0·33	8·48*	0·28
Zinc (passivated)	8·07	0·20	8·55*	0·30
Tin-Zinc Alloy (50% Tin)	7·29	0·28	7·78	0·32
Tin-Zin Alloy (50% Tin) (passivated)	7·69	0·28	7·66	0·32
Tin-Zinc Alloy (80% Tin)	7·28	0·29	7·66	0·44
Tin-Zinc Alloy (80% Tin) (passivated)	8·02	0·18	8·02	0·24
Cadmium	7·14	0·27	7·18	0·36
Cadmium (passivated)	7·00	0·32	7·14	0·20
Tin	8·15	0·24	7·87*	0·34

* Exposed for 44 months as against 18 months for others.

TABLE V.—CUP-DEPTHS (MM.) TO FRACTURE IN AVERY SHEET METAL TESTER OF SHEETS EXPOSED AT PERIVALE. EACH VALUE IS DERIVED FROM 12 TESTS.

Coating.	0·0003 in. coatings exposed for 25 months.		0·0005 in. coatings exposed for 44 months.	
	Mean	Standard Deviation	Mean	Standard Deviation
Zinc	8·92	0·39	8·82	0·23
Zinc (passivated)	8·57	0·28	8·80	0·29
Tin-Zinc Alloy (50% Tin)	8·42	0·26	7·99	0·35
Tin-Zinc Alloy (50% Tin) (passivated)	8·27	0·20	8·03	0·29
Tin-Zinc Alloy (80% Tin)	7·77	0·31	7·60	0·31
Tin Zinc Alloy (80% Tin) (passivated)	8·18	0·25	7·68	0·26
Cadmium	7·43	0·30	6·64	0·32
Cadmium (passivated)	7·47	0·31	6·72	0·37
Tin	8·26	0·24	8·08	0·24

TABLE VI.—CUP-DEPTHS (MM.) TO FRACTURE IN AVERY SHEET METAL TESTER OF SHEETS EXPOSED AT HURST CASTLE. EACH VALUE IS DERIVED FROM 12 TESTS.

Coating.	0·0003 in. coatings exposed for 29 months.		0·0005 in. coatings exposed for 45 months.	
	Mean	Standard Deviation	Mean	Standard Deviation
Zinc	8·49	0·42	8·30	0·19
Zinc (passivated)	8·72	0·30	8·13	0·32
Tin-Zinc Alloy (50% Tin)	8·99	0·15	8·79	0·45
Tin-Zinc Alloy (50% Tin) (passivated)	8·65	0·38	8·28	0·31
Tin-Zinc Alloy (80% Tin)	8·44	0·38	7·85	0·27
Tin-Zinc Alloy (80% Tin) (passivated)	8·35	0·24	8·19	0·37
Cadmium	8·06	0·29	8·48	0·34
Cadmium (passivated)	8·14	0·17	8·25	0·35
Tin	7·89*	0·19	7·94*	0·19

* Tin-coated panels exposed for 18 months only.

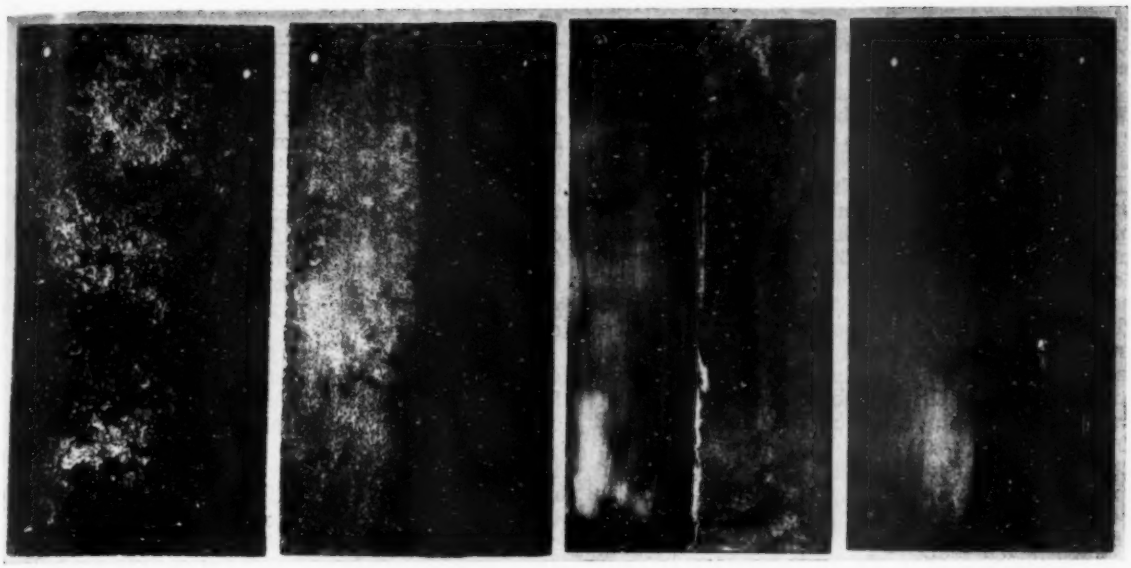

Fig. 4—Panels after test in intermittent salt spray. The coatings are (left to right): None, Zinc, 80/20 Tin-Zinc, Tin. The corrosion product has been removed from the whole of the bare steel panel and from the left-hand halves of the others.

The main function of the passivation treatments is to restrain disfigurement of the coatings in indoor corrosion by mild condensation or handling. It appears that they will not usually be worth while to delay rusting in out-of-doors exposure.

II.—Cupping Tests

When the panels were withdrawn, an attempt was made to assess their deterioration by means of a cupping test. From every panel, 12 test specimens were taken from 2-in. strips cut with centre lines 2 in. from each long edge. The upper, fully weathered, surface was made the convex one in all tests. The results from any one panel had some scatter, quite evidently due in many instances to different degrees of rusting between the individual test-pieces, but sometimes without visible reason. Nevertheless, there were some very distinct differences between the sets of results for different panels.

The mean and standard deviations of the results for the 0·0003-in. and 0·0005-in. coatings are presented in Tables IV–VI. The 0·001-in. coatings have not yet been withdrawn. The mean of 20 determinations of cup-depth on uncoated, unexposed sheet was 8·72 mm. with a standard deviation of 0·25 mm.

The size of the standard deviations is in many instances too large to permit a reliable estimate of the orders of merit by simple inspection. An attempt has been made to assess the significance of the differences by calculating the "standardised error" or "t function" of the difference between pairs of the mean values obtained for the various coatings.

The orders of merit then emerging after disregarding differences producing a value of the "t function" which could arise by chance more than one in a hundred times are shown in Table VII. In doubtful cases, such as when the results for A are significantly better than those for B, but the values for C are not significantly different from those of either A or B, A has been ranked as 1, B as 3 and C as 1–3.

The general indications given are in fair agreement with those given by visual observation. Some differences are given greater emphasis, e.g. the inferiority of the cadmium coatings and the good performance of the tin coatings at Oldham and Perivale. For cadmium, the more rapid spread of rust, once it has begun, and the more rapid failure on the under-side of the panels is reflected better in the cupping test results than by the recorded observations of the upper surface. For tin, the visual observations were difficult to interpret because of the early appearance of rust and its very slow development, but the good cupping-test values show that deterioration of the steel was truly slow. They indicate too that there was no injurious pitting and this was confirmed by examination with the microscope of sections cut from Oldham panels. On the other hand the tin coatings from Hurst Castle gave poor results in the cupping test, reflecting the undoubted pitting taking place there.

Discussion

The results of visual observation and of cupping tests made on the exposed panels agree in indicating that for out-of-doors exposure in urban and suburban districts the order of merit of the coatings for the range of thickness tested is: zinc, 50/50 tin-zinc, 80/20 tin-zinc, cadmium, with tin occupying the lowest position or a

TABLE VII.—ORDERS OF MERIT BASED ON CUPPING TEST RESULTS AFTER EXPOSURE.

Coating	Order of Merit					
	Oldham		Perivale		Hurst Castle	
	0·0003 in.	0·0005 in.	0·0003 in.	0·0005 in.	0·0003 in.	0·0005 in.
Zinc	4	1	1	1	2–6	3
Zinc (passivated)	1	1	2	1	2	3–8
Tin-Zinc Alloy (50% Tin)	6	4–6	2–3	3	1	1
Tin-Zinc Alloy (50% Tin) (passivated)	4	6	3	3	2–6	3
Tin-Zinc Alloy (80% Tin)	6	6	7	6	2–6	8
Tin-Zinc Alloy (80% Tin) (passivated)	1	4	3	6	6	3
Cadmium	6–9	8	8	8	7	1–3
Cadmium (passivated)	9	9	9	8	7	3
Tin	1	3	3	3	9	8

high position immediately after zinc depending on whether judgment is based on earliness of rust appearance or on protection of the steel from loss of strength. In the marine exposure, the results were quite different, the 50/50 tin-zinc alloy being best, followed by zinc, not far ahead of cadmium and the 80/20 tin-zinc alloy, and tin much the worst.

These results differ somewhat from those given in laboratory salt-spray and humidity chamber tests previously reported. The tests of Angles and Kerr[3] indicated that tin-zinc coatings with 50–80% tin delayed rusting of steel for much longer in the salt-spray test and a little longer in a "tropical humidity" test than zinc and cadmium. Halls[4] found a similar result in salt-spray, but found that zinc delayed rusting a little longer than the alloy coatings in a humidity test. The reports of both sets of tests stress that the formation of bulky white corrosion product is severe with zinc but only slight with the alloy. This feature was only evident in the outdoor tests on the under-sides of the panels and, even there, was less marked than in the more continuously humid laboratory test conditions.

The marine exposure gives, as might be expected, results most close to those of the salt-spray test but, even in this, the relative behaviour of the zinc coatings was inferior to that predicted by the accelerated test. On the other hand, the behaviour of panels which were intermittently immersed in the sea gave a result rather similar to that of the salt-spray test. In these tests 12×8-in. panels held in wooden frames were fixed at a half-tide position in Torquay Harbour. Tin-zinc alloy coatings easily outlasted zinc coatings of similar thickness and were a little inferior to cadmium.

It seemed possible that a relatively poor performance of zinc might be associated with almost continuous wetting with salt water and further salt-spray tests were carried out in which the test panels were allowed to dry for 16 hours out of every 24, with a washing down with water between spraying and drying. Even then the zinc coating was much inferior to tin-zinc, both in regard to the formation of white corrosion product of the coating and to the delaying of rusting of the steel. The examples from this test illustrated in Fig. 4 were 0·0005-in. coatings exposed to spray of 3·5% sodium chloride solution for 8 hours daily over a period of 250 days. It will be seen that the condition of the steel protected by 80/20 tin-zinc alloy is unchanged although an unprotected steel sample 0·048 in. thick was perforated and a zinc-coated sample severely corroded. In similar tests, using water spray in a chamber with its atmosphere polluted by burning sulphur-enriched coal-gas, the tin-zinc coatings again proved superior to zinc coatings, a result contrary to the effect of full exposure to weather in any situation, but consistent with all other results obtained in tests involving long periods of continuous humidity. Although the results of laboratory tests conflict with those obtained out-of-doors, their indication that the tin-zinc coatings behave relatively well as compared with zinc in humid conditions is itself a useful one.

Cadmium was not included in these laboratory tests. Its lower performance, relative to zinc, in industrial atmospheres, as compared with its behaviour in salt-spray is well known and no evidence obtained in the outdoor test conflicted with expectations from the earlier laboratory tests.

The practical importance of the weathering tests lies largely in the comparison between tin-zinc and cadmium. The general use of zinc coatings for protection of steelwork fully exposed to the weather is well established and the tests described show that neither tin-zinc nor cadmium is a serious challenger to zinc in this field. For some uses however, chiefly in the electrical and radio industries, the relatively poor solderability of zinc and its liability to form bulky white corrosion product in condensation of moisture make it a less desirable coating than either tin-zinc alloy or cadmium. Both of these coatings are readily solderable, using non-corrosive resin fluxes, they are much less prone than zinc to form loose bulky corrosion products and may even give better protection against rusting in continuously humid conditions.

Now that long period weathering confirms the results of earlier laboratory accelerated tests that tin-zinc coatings can give protection at least equal to and often better than that given by cadmium coatings, the alloy may be preferred. Apart from relative availability, which has favoured the use of the alloy for some time, the cost of using the alloy is lower. Experience with large-scale industrial production in a semi-automatic plant over a period of 9 months in 1947-48 showed that the relative cost of materials and heating of electrolyte for equivalent output were tin-zinc, 1 to cadmium, 1·68[6]. Other costs were the same for the two coatings.

The better performance of the 50% tin alloy out-of-doors as compared with the 80% alloy may in practice not be exploited. The operating conditions for deposition of the 80% alloy are less exacting and a change-over from cadmium deposition to alloy deposition requires no change of plant. Moreover the 80% alloy is less susceptible than the 50% alloy to the formation of bulky white corrosion product in humid conditions.

The good protection obtained with tin in full weathering in urban and suburban situations will probably have no direct practical application because of the relatively high cost of the coating. The result is, however, useful in demonstrating the error of those who still say that a tin coating will always accelerate the penetration of steel exposed at pores in it. (Even in salt spray this may not occur, as is shown in Fig. 4, where the steel panel tested uncoated is seen as perforated in the time taken to produce insignificant pits on the tin-coated panel.) The result also has a practical bearing in that the checking of galvanic corrosion at the pores by the presumed high electrical resistance of the pore-channels through the coatings may also be achieved with much thinner tin-coatings by painting them. A tin coating 0·00005 in. thick where it receives the additional protection of paint is very resistant to rusting, even in a marine situation. Examples of this effect for the Hurst Castle exposure site have been shown elsewhere[7].

Summary and Conclusions

For full exposure to the weather in urban or suburban locations, the protection given to steel by coatings of equal thickness diminished in the order zinc, 50/50 tin-zinc alloy, 80/20 tin-zinc alloy, cadmium. By the sea, an alloy with 50% tin was rather better than zinc and cadmium, but an 80% tin alloy was inferior to zinc and no better than cadmium.

Tin coatings, although permitting the early appearance of rust, were very effective in preventing serious damage

6 P. J. Miller and J. W. Cuthbertson, *Metal Finishing* (1949), **47**, 44.
7 S. C. Britton, *J. Oil and Colour Chem. Ass.* (1950), **33**, 125.

to the steel in urban and suburban locations, but failed rather badly in marine conditions.

In tests involving more continuously humid environments, the tin-zinc alloy coatings were better than zinc both in producing a less bulky white corrosion product and in preventing rusting of the steel.

The conclusions drawn from visual observation of the panels exposed to the weather were confirmed and, to some extent, increased in clarity by cupping-tests carried out after rusting had made some progress.

It is considered that although tin-zinc alloy coatings cannot challenge zinc coatings for use solely as a protection against the weather, they have advantages for use in situations of prolonged high humidity and when a coating easily solderable with a non-corrosive flux is required. For these conditions they offer an effective alternative to cadmium, providing at least an equal service at lower cost.

Acknowledgment

The tests described were organised at the Tin Research Institute and the authors are indebted to the International Tin Research and Development Council for permission to publish the results. Exposure sites were made available by the kindness, at Oldham, of Messrs. Cossor Radar, Ltd. and Messrs. S. Dodd & Sons, Ltd., and, at Torquay, of the Mayor and Corporation. Mr. T. R. A. Windeatt co-operated in the mounting and inspection of panels at Torquay.

Some Recent Advances in Theoretical Metallurgy

Earlier this year a series of lectures on Some Recent Advances in Theoretical Metallurgy was given at the Technical College, Bradford, by members of the College staff. The aim of the course was to present recent theoretical developments with particular reference to their practical applications. A condensed account of the subject matter of the lectures, prepared by the lecturers concerned, is presented here.

The Atomic and Electronic Structure of Metals

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MODERN theories of solids provide a picture of the constituent atoms and the way in which they are built up into macroscopic materials. They also provide interpretations of mechanical and physical properties and reveal the factors determining metallic or non-metallic behaviour of elements.

Atoms consist of a nucleus composed of protons and neutrons, surrounded by electrons. The number of protons determines the net nuclear charge and the number and arrangement of electrons in the neutral atom. These, in turn, determine the chemical properties and the position of the atom in the periodic table. Information regarding the energy levels of the extra-nuclear electrons is obtained from a study of atomic spectra, which result from electronic energy changes in atoms, leading to emission of light. The interpretation of atomic spectra thus requires an appreciation of the nature of light.

Diffraction experiments suggest that the propagation of light is governed by the laws of wave motion. The interaction of light with atoms, as in the photoelectric effect, suggests the propagation of energetic particles (photons) guided by waves, the property determining the ability of a photon to expel an electron from a metal, that is, determining the energy of the photon, being its frequency. The energy e of a light photon is given by the product of the frequency and a fundamental constant (Planck's Constant h).

The energies necessary to remove an electron from isolated atoms (ionisation potentials) can be obtained from optical spectra, and the corresponding energies for removal from a solid metal (work functions) can be obtained from the photoelectric effect. In general, metals have low ionisation potentials while for non-metals this value is high.

X-ray examination of solids has revealed their internal structure and provided a fundamental basis for their classification. The four main extreme types of solid are ionic crystals, molecular crystals, valency crystals and metals. Ionic crystals are formed by electron transfer between the component atoms, valency crystals are formed by the sharing of electron pairs between neighbouring atoms (covalent links) extending throughout the crystal lattice, and molecular crystals are loosely bound aggregates of molecules, held together by weak intermolecular forces.

Metals exhibit an internal arrangement in which the predominant feature is a high lattice-co-ordination number. The number of nearest neighbours is 8 in the body-centred arrangement and 12 in either of the arrangements corresponding to closest packing of equal spheres, namely, the face-centred and close-packed hexagonal lattices. Malleability and ductility are explained in terms of ease of deformability of such arrangements without cleavage.

The cohesion of metals, which is large, is thus due to bonding of a different nature from that found in other types of solid. A useful, if elementary, picture may be obtained by imagining that the valency electrons are free to move in the body of the metal, which is a lattice of positive ions (atoms stripped of valency electrons). The stability of the lattice can be regarded as due to the attraction between the electron gas and the positive metal ions, which balances the mutual repulsion of the latter. Cohesion is large as shown by the high surface energy of metals and their high melting and boiling points.

The Drude-Lorentz theory of metals imagined conductivity as due to accelerated motion of the mobile electrons under the influence of an applied potential and opposed by random thermal motion of the electrons. Resistances calculated were of the right order but increased linearly with temperature, which was not predicted by the theory. A further weakness of the simple theory was the insignificant electron contribution to heat capacity found in practice. This implied that normally only a very small fraction of the valency electrons could be regarded as free to acquire increasing thermal energy with rise in temperature.

These facts are explained by the wave-mechanical theory, which is founded on the observation that a beam of electrons obeys wave propagation rules similar to those observed for light photons, forming a concentric ring diffraction pattern after passage through a thin metal foil. The apparent wavelength λ of the associated wave nature (De Broglie wave) depends on the momentum p. Since $\lambda = h/p$, wave nature can only be demonstrated for small particles, for which λ is sufficiently large to give a diffraction pattern with the smallest grating available (atomic lattice).

A single electron would not generate the whole diffraction pattern and its behaviour could only be stated in terms of a probability distribution. Similarly, in an atom, we can only give a probability distribution for an electron. The De Broglie waves of electrons confined to move within fixed limits (bound electron in an atom or metal lattice) cannot assume any given frequency. Their frequencies may take only certain values analogous to the fundamental and harmonics (standing waves) in a stretched elastic string. Since energy depends on frequency, electrons may have only whole number multiples of certain minimum energies. Each energy level may be occupied by only two electrons which have equal and opposite internal (spin) energies (Pauli Principle). For atoms, these levels are well characterised from studies of optical spectra. They correspond to three-dimensional wave patterns with spherical nodes (s-electrons), nodal plane (p-electrons), two nodal planes (d-electrons).

In a metal, electrons occupy available levels pairwise, filling a band or zone of energy levels. Only those in the uppermost levels can normally acquire additional energy thermally or under an electrical potential. Only a small fraction at the top of the band are normally available to jump to unoccupied levels and thus the electron gas makes no significant contribution to heat capacity. The closely spaced energy levels are, however, broken up into permitted bands (Brillouin zones) separated by forbidden regions of energy, due to diffraction by the lattice. The electrons move in a periodically fluctuating potential field low in the neighbourhood of atoms and high between them. Closest approximation to uniform potential corresponds to a small charge on the lattice ions and it is significant that elements in the early periodic groups are metals.

Brillouin zones may be widely separated or overlap, completely filled or partly filled, depending on lattice arrangement (unit cell) and valency. The metallic or non-metallic character depends on the relative distribution and filling of the Brillouin zones. Phase transitions in binary alloy systems occur at critical electron/atom ratios, which are determined by the availability and filling of zones in the unit cells corresponding to the respective phases.

Modern theories present a unified picture of all types of solid, interpreting particularly clearly the characteristic properties of metals and providing a theoretical explanation of the Hume-Rothery rules.

Applications of Electronic and Atomic Theories to the Structure and Properties of Alloys

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CONSIDERABLE interest is shown by metallurgical students in the theories associated with the inner structure of metals. This interest should receive encouragement even if the subject is outside the daily activities of students in industry. Only in this way can one keep abreast of developments in alloys that may result from the electronic and atomic theories. In the following article, it is impossible to do more than indicate certain of the more important applications of these theories to metallurgy. A brief account will be given of the Hume-Rothery Rule, the electro-negative valency effect, the relative valency effect, the form and position of phase boundaries and a reference is made to superlattices. A bibliography is given at the end of this article for purposes of reference and further reading.

The electron groupings, for the " free " atoms, so as to indicate their association with the principal and secondary quantum numbers, are of special importance as regards the properties of metals and the application of the atomic theories to structural metallurgy. In particular, it is the outer (valency) group of electrons which is considered responsible for metallic properties. Thus to take one prominent element, copper, it is the one electron in the four-quantum shell which is the valency electron and responsible for metallic properties.

Atomic Number:	Element:	n =	1	2		3			4			
		l =	0	0	1	0	1	2	0	1	2	3
29	Cu		2	2	6	2	6	10	1			

These considerations of electron groupings form, for example, the basis of the Hume-Rothery Rule which relates the number of valency electrons to the number of atoms for metallic systems that form solid solutions. It was found that the β-phases in certain systems had the common characteristic that they corresponded to a ratio of three valency electrons to two atoms. Thus in CuZn (*ca* 50 atomic % Zn) there are two atoms and three valency electrons, one electron from the copper atom and two from the zinc atom. In Cu_5Sn (16·7 atomic % Sn) there are six atoms and nine valency electrons, four from the tin and one from each copper atom.

It is convenient at this stage to consider briefly the mechanical properties of primary solid solutions. They possess, excellent toughness and ductility combined with higher tensile strength than that of the major

constituent metal above, besides being relatively resistant to atmospheric and chemical corrosion as a result of their one-phase structure. The α-brasses and also bronzes are thus important in this respect, and the changeover to the hot-rolling of 70 : 30 brass where, previously, cold-working was general, also serves to show further the favourable properties of solid solutions. "Monel" metal is an important industrial alloy of the nickel-copper range of solid solutions, and the "Nimonic" alloys, which possess high creep-resistance under service conditions, are associated with the α-solid solution of chromium in nickel.* The 18% Cr, 8% Ni (austenitic) steels are further examples of solid solutions of great industrial use. Application of the electronic and atomic theories enables prediction to be made regarding the probability of particular metals forming solid solutions.

Brillouin-zone diagrams are of much interest. For example, they show why metals are good conductors of electricity, because where overlapping of the Brillouin zones occurs the flow of electrons (associated with an electric current) can take place due to there being unoccupied electron states, whereas a gap between zones indicates that further energy cannot be taken up and, in consequence, there is no flow (insulation). The Brillouin-zone diagram relates the number of electronic states to the energy of the electrons.

The most important method of determining the presence of phases, and their boundaries, in alloy systems is by use of the metallurgical microscope. Another method of determining the position of phase boundaries in alloy systems is by determining the free energy of two solid phases at a given temperature and relating it to their atomic composition. When the two phases are in equilibrium with one another the total free energy must be a minimum for any internal change of the whole system (the second law of thermodynamics). By drawing a common tangent to the two curves representing the variation of free energy of the two phases with composition, it is possible to determine the boundaries within which both phases are present together, whereas outside either boundary only one of the phases is present.

Superlattices in solid solutions appear as an ordered arrangement of particular atoms in the lattice. Where there is no ordered arrangement of particular atoms, only a random "substitutional" solid solution may be regarded as existing. Many instances of superlattice structures occur. A superlattice occurs in the industrially important brass containing the β-phase, in which the change from order to disorder, for example on heating the alloy, occurs in the neighbourhood of 460° C. Fascinating results regarding the superlattices in the iron-aluminium system have been described by Bradley and Jay. Using the X-ray diffraction method of analysing the internal structure, they showed how the iron atoms and the aluminium atoms take up different positions in the lattice as the composition of the iron-aluminium alloy varies.

Regarding primary metallic solid solutions, X-ray diffraction has shown that such a solution has the same structure as the parent metal from which it is formed and two possibilities arise. First, there is the formation of substitutional solid solutions and, second, interstitial solid solutions. The former are the more general. However, an important example of the interstitial type is that of carbon in γ-iron, which forms the basis of the steels and, as one would expect, this type occurs when one atom is very much smaller than the other. Again, there is the marked influence on the mechanical properties of small amounts of elements which dissolve interstitially. In ternary alloys, both types of solid solution may exist together as in austenitic manganese-steel, where there is a substitutional solid solution of manganese in iron and an interstitial solid solution of carbon in iron.

The application of electronic and atomic theories in metallurgy has resulted in the formulation of (*a*) the idea of atomic "size-factor," (*b*) the electro-negative valency effect and (*c*) the relative valency effect, besides affording a valuable insight into the form of liquidus and solidus curves. About 1934, Hume-Rothery, Mabbott and Channel-Evans put forward the hypothesis that when the atomic diameters of solvent metallic element and solute metallic element differ by more than about 14–15% of that of the solvent, the "size-factor" is unfavourable and the solid solution is very restricted, whilst when the atomic diameters are within this limit the "size-factor" is favourable, and considerable solid solutions of the metals may be formed, subject to certain restrictions. (The atomic diameter is arbitrarily taken as the closest distance of approach of the atoms in the crystal structure of the element.) Without giving actual atomic diameters, we may note, for example, that zinc is in the favourable zone of "size-factor" for copper and silver. Metallurgical investigations have shown that there is a wide range of solid solutions of zinc in copper and in silver. However, solid solutions are not always formed when "size-factors" are favourable so that the conception of "size-factor" is most useful as a kind of preliminary test to indicate whether two metals are unlikely to form solid solutions. When "size-factor" restricts the formation of a wide solid solution range, the solubility limit of the restricted solid solution increases with temperature so that alloys of this kind may be expected to show "precipitation-hardening."

The electro-negative valency effect is concerned with the tendency of two or more metals to form stable intermetallic compounds at the expense of primary solid solutions, even though the "size-factor" is favourable. Generally speaking, the more electro-negative the solute element and the more electro-positive the solvent (or vice-versa), the greater is the tendency for the formation of stable intermediate compounds. Thus the electro-negative character of phosphorus, arsenic, antimony and bismuth is pronounced yet perhaps not well marked, and in consequence slight solid solutions of these elements may be formed provided that the solvent metal is not too electro-positive in nature. Again, stable compounds precipitate to give the phenomenon of "age-hardening," as with the compound Mg_2Si in the ageing of aluminium alloys containing Mg and Si.

Relative valency effects are concerned with whether the tendency of two metals to form solid solutions is necessarily reciprocal. The hypothesis is that, other things being equal, a metal of lower valency may be regarded as being more likely to dissolve one of higher valency than vice-versa. The rule appears to be a very general one for alloys of the univalent elements with those of higher valency.

Regarding the form of liquidus and solidus curves, it may be shown that, when copper (and silver) is the solvent of certain metals, increasing valency of the solute results in a more restricted solid solution and a steeper

* The high creep strength is, however, also associated with certain other necessary conditions.

fall in the liquidus and solidus curves. It is at once apparent that a definite valency effect exists. The same general effect of valency also applies for the position of the solid-solubility curves, and the effect also applies to some of the physical properties of the solid solutions. Thus, in general, the increase in electrical resistance of copper, silver or gold, caused by one atomic percentage of certain different elements in solid solution, increases with the valency of the solute. Examination of various metallic systems suggests that the depression of the solidus curve increases with the difference in "size-factor" and valency of solvent and solute, and that, in general, the solidus curves are much more affected by these factors than are the liquidus curves. Also, if certain equilibrium diagrams are constructed in terms of electron concentration, the solidus and solid-solubility curves are roughly superimposed.

This account of applications of the electron and atomic theories to metallurgy, which has dealt with the valuable mechanical properties of solid solutions and related ideas of "size-factor," electro-negative valency effect, relative valency effect, should be of interest to persons requiring a short introduction to the conceptions that have been proved necessary in understanding the formation of alloys and the prediction of their properties.

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Some Applications of Thermodynamics to Process Metallurgy

By W. R. Moore, Ph.D., B.Sc., F.R.I.C.

Senior Lecturer in Physical Chemistry

TWO methods—the kinetic and the thermodynamic—may be used in the interpretation of chemical and physical processes. Starting with a picture of the behaviour of individual atoms or molecules, the kinetic method permits, in many cases, the interpretation of such processes. It does not, however, generally permit the prediction of chemical reactions or such physical phenomena as solution or miscibility. Such predictions may be made using the thermodynamic method which deals with the movement of any given element from one phase or state of combination to another.

Two factors determine the tendency to a chemical or physical change, these being :

(*a*) The heat factor, measured generally by the heat of reaction, $\triangle H$, and determining the tendency of the system to move to a position of lower energy.

(*b*) An order factor arising from the tendency of all matter to pass to a state of more random arrangement. This opposes the first factor and increases with temperature. It is measured by $T \triangle S$ where $\triangle S$ is the entropy change when the process occurs, entropy being a measure of the randomness or degree of disorder of the system. The net tendency of a system to change may be measured by the change in Gibbs Free Energy $\triangle G$ which is related to the heat and order factors by

$$\triangle G = \triangle H - T \triangle S$$

The value of $\triangle G$ is a measure of the tendency of the process to occur, and if the process occurs $\triangle G$ must be negative. Much of thermodynamics is concerned with the evaluation of $\triangle G$, permitting the prediction of possible chemical or physical processes.

For the comparison of different chemical reactions it is convenient to use the standard Gibbs Free Energy change $\triangle G^\circ$, the value of $\triangle G$ for the reaction when the reactants are initially at unit activity and the resultants are finally at unit activity. For a reaction represented generally by

$$aA + bB = cC + dD$$

the equilibrium constant K is given by

$$K = \frac{a_C^c \times a_D^d}{a_A^a \times b_B^b}$$

where the a's are the activities of the reacting substances at equilibrium, and are measures of the tendency of each substance to react. It may be shown that

$$-\triangle G^\circ = RT \ln K$$

The equilibrium constant K varies with temperature and if this is taken into account it may be shown that

$$-\triangle G^\circ = \triangle H_\circ + \alpha T \log_e T + \frac{\beta T^2}{2} + \frac{\gamma T^3}{6} + IT$$

where α, β, and γ are constants depending on the heat capacities of the reacting substances, $\triangle H_\circ$ is given by

$$\triangle H = \triangle H_\circ + \alpha T + \frac{\beta T^2}{2} + \frac{\gamma T^3}{3}$$

and $\triangle G^\circ$ can therefore be obtained from a knowledge of α, β, and γ and $\triangle H$, the heat of reaction at the absolute temperature T. I is a constant which can be obtained from a knowledge of $\triangle G^\circ$ at one temperature.

While the expression for the variation of $\triangle G^\circ$ with T is complicated a plot of $-\triangle G^\circ$ against T for a chemical reaction is nearly linear. Such plots have been used by Dannatt and Ellingham[1] in studies of roasting and reduction processes and by Richardson and Jeffes[2] in studies of slag formation. The construction and use of such plots has been described by Ellingham[3]. At a given temperature the larger the value of $-\triangle G^\circ$ the greater the likelihood of the reaction occurring. Such plots show that a reaction with a large value of $-\triangle G^\circ$ will tend to drive backwards one with a smaller value. Thus in the reactions

$$4/3\ Al + O_2 = 2/3\ Al_2O_3$$

and

$$2\ Mg + O_2 = 2MgO$$

the latter reaction has the larger value of $-\triangle G^\circ$ and thus Mg can remove oxygen from Al_2O_3 giving MgO and Al. Plots giving data for many such oxidation reactions

may be used and since data for the oxidation of carbon and the formation of water may be included, many metallurgical reactions can be predicted.

If the reaction can be carried out in an electrolytic cell $\triangle G^\circ$ can be related to the E.M.F. of the cell by

$$-\triangle G^\circ = nFE$$

where n is the number of charges involved in the reaction, F the Faraday and E the E.M.F. Cells of the Daniell type with molten halides as electrolytes have been used by Lorenz[4] in the study of displacement reactions between metals. Baur[5] has used such cells in the study of the oxidation of metals. They have also been used by Rose, Davis and Ellingham[6] in the study of reduction processes.

Values of $\triangle G^\circ$ may be obtained, in certain cases, from values for related reactions. Thus the value for the water gas reaction,

$$CO_2 + H_2 = CO + H_2O$$

can be obtained from values for the reactions

$$2H_2O = 2H_2 + O_2 \text{ and } 2CO_2 = 2CO + O_2$$

$\triangle G^\circ$ may also be obtained, if $\triangle H^\circ$ is known, from values of $\triangle S^\circ$, calculated from thermal data using the third law of thermodynamics.

Many of the properties of liquid mixtures of metals or substances of metallurgical interest can be interpreted by use of partial molar Gibbs Free Energies or activities of the components. For a mixture of 2 components, (1) and (2), if G is the Gibbs Free Energy of the mixture,

$$G = n_1\bar{G}_1 + n_2\bar{G}_2$$

where n_1 and n_2 are the number of gm-atoms of the two components and G_1 and G_2 their partial molar Gibbs Free Energies. The condition of equilibrium between two phases is that the value of G for a component common to both phases must be the same in these two phases. $\bar{G}$ may also be defined by

$$\bar{G} = G_\circ + RT\log_e a$$

where G_0 is the Gibbs Free Energy of one gm-mol or gm-atom of the component and a its activity defined by

$$a = Nf$$

where N is the mol or atom fraction and f the activity coefficient which is a measure of the deviation of the component from ideal behaviour ($a = N$; $f = 1$). Activities are also given by

$$a = p/p_\circ$$

where p is the partial vapour pressure of the component above the mixture and $p_\circ$ its saturation vapour pressure at the same temperature.

The last relationship provides a means of determining activity. The activity of a component may also be obtained from phase diagrams, making use of the facts that the activity of a component is the same in two phases in equilibrium and that the difference in Gibbs Free Energy between the liquid and solid states of a pure solid component can be calculated from heats of fusion. This method has been used to determine the activity of iron in iron/iron sulphide.[7] Activities may also be obtained by use of concentration cells in which the electrodes are alloys involving the same metals but of differing composition and the electrolyte is a fused salt containing the ion of the more electropositive metal. If E is the E.M.F. of the cell and n the valency of the more electropositive metal, then

$$-nFE = RT\log_e(a_1/a_2)$$

If one activity is known or corresponds to a dilute solution, when $a = N$, the other is readily calculated.

$\triangle G^\circ$ versus T plots also, in certain cases, provide a means of determining activities. Thus, for the reaction

$$2FeO + SiO_2 = Fe_2SiO_4$$

such plots give $-\triangle G^\circ = 5{,}800$ cals at 1,600° C. From this, the equilibrium constant K (given by $-\triangle G^\circ = RT\log_e K$) can be found. Since K is given by

$$K \times a_{Fe_2SiO_4}/a^2_{FeO} \times a_{SiO_2}$$

if the activities of two of the reacting substances are known that of the third can be calculated.

In general, the larger the activity or activity coefficient of a component in a mixture, the lower the miscibility and the easier its removal by such processes as distillation. The high activity of zinc is made use of in the purification of desilverised lead containing *ca* ½% zinc by vacuum distillation. Small activities or activity coefficients imply high miscibility or even compound formation as in the system mercury-sodium. Metals such as aluminium, silica and titanium, important in the deoxidation of steel, exhibit small activity coefficients in liquid iron.

Addition of a third component to a binary mixture may increase or decrease the activities of the original components. Thus the activity of sulphur is increased by addition of carbon to steels leading to the formation of two liquid layers, the bulk of the sulphur occurring in the ferrous sulphide layer. Deoxidisers such as chromium and vanadium decrease the activity of oxygen in molten iron. Addition of lime to the silica-ferrous oxide system increases the activity of ferrous oxide so that the slag finally contains less than ½% iron as silicate, more ferrous oxide being reduced by carbon monoxide to iron. The low activity coefficient of lead monoxide in mixtures with silica is made use of when lead is removed from copper by the addition of silica at the end of the oxidising period in the refining of copper. Phosphorus may be removed from molten iron by a lime-rich slag which holds the phosphorus as a phosphate of low activity.

The application of thermodynamics to metallurgical problems thus provides an alternative and often very fruitful method of approach, revealing much of the general pattern of chemical and physical processes and permitting the prediction of the course of such processes.

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First Holders of Athlone Fellowships Arrive

THE first Canadian engineering graduates to receive awards under the new Athlone Fellowship scheme have arrived in this country. Beginning this year, 38 Athlone Fellowships are to be awarded each year by H.M. Government to enable Canadian engineering graduates to take post-graduate training in the United Kingdom. Fellowships will cover the cost of transport, fees and maintenance and be tenable normally for a maximum period of two years. They will be available for one of the following options: (a) Works training in one or more approved industrial organisations; (b) post-graduate study in a United Kingdom university or college or research establishment; (c) a combination of (a) and (b); or (d) research in a United Kingdom University leading to the degrees of M.Sc. or Ph.D. 28 of the Fellowships will be awarded annually to newly graduated candidates, and 10 to graduates from industry.

Catalytic Gas Purification

DEVELOPED in America by Baker & Co., Inc., the Deoxo process of catalytic gas purification is being introduced into this country by Baker Platinum, Ltd., London, associates of the American company. The process has proved highly beneficial in many applications in American industry.

Many industrial processes require gases which are virtually free from oxygen. In the metallurgical field, this is particularly true in certain bright-annealing operations and in powder metallurgical processes. Commercial hydrogen or nitrogen may contain some oxygen, up to 0·3% or even more, which is harmful in many cases. The older method of removing oxygen from hydrogen, nitrogen, etc., involved passing the gas over copper chips heated to 600°–650° C. This removed practically all the oxygen, the last traces being removed (down to a low figure), by passing over ferro-silicon, ferro-manganese, or ferro-titanium heated to 900°-1000°C.

In the Deoxo system oxygen is removed from hydrogen by passing the gas over a specially prepared precious metal catalyst which brings about combination of the oxygen with twice its volume of hydrogen to form water vapour. This reaction will take place at room temperature: this is a significant feature of the process. The resultant water vapour can be removed by any standard dehydrator. The heat generated by the catalytic reaction amounts to a temperature rise of some 10° C. for each 0·1% of oxygen removed.

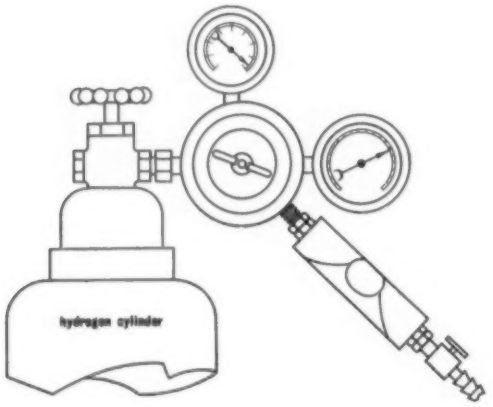

Diagram showing gas connections for Deoxo purifier model 5–50.

After passing successively through the Deoxo tower and a regenerative dryer of adequate capacity, less than one part per million of oxygen remains. The hydrogen is then sufficiently pure to reduce oxides of chromium, and to sinter 18 : 8 stainless steel compacts, Alnico : 80 : 20 nickel chromium, and similar alloys. The H_2O/H_2 ratio for equilibrium with iron are such that there is no difficulty in bright annealing iron in hydrogen of normal purity, but at elevated temperatures, oxygen and water vapour impurities in hydrogen will form selective oxides with chromium, manganese, silicon, vanadium, zinc, aluminium, beryllium, titanium and certain other metals. Accordingly, when metals containing these constituents are being annealed in a hydrogen atmosphere it is sometimes necessary to purify the incoming gas.

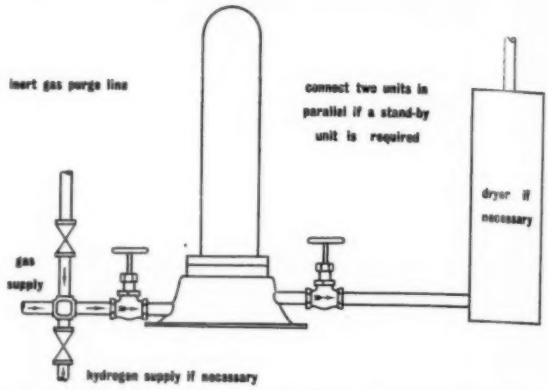
inert gas purge line
connect two units in parallel if a stand-by unit is required
dryer if necessary
gas supply
hydrogen supply if necessary

Diagram showing gas connections for Deoxo purifier model 200–50.

From the nature of the reaction it will be clear that unwanted oxygen or hydrogen can be removed from inert gases such as nitrogen, argon, helium, neon, carbon-dioxide, and saturated hydrocarbons, provided there is always a ratio of two volumes of hydrogen to one volume of oxygen in the gases to be treated. If one or other of the gases is not present in the free state, it must be added to the gas stream ahead of the purifier in the correct volume necessary to effect a complete combination.

Experience to date concerns, mainly, the removal of oxygen from hydrogen, but development is in progress on units to provide for the catalytic combination of: (*a*) oxygen and hydrogen in the presence of carbon monoxide; (*b*) oxygen and carbon monoxide to form carbon dioxide in gases that do not contain hydrogen; (*c*) carbon monoxide in hydrogen to form carbon dioxide; (*d*) carbon monoxide and hydrogen to form methane and water vapour.

The Deoxo units are of simple, compact and rugged construction and there are no operating or maintenance expenses. Furthermore no cooling water is required, and no electrical current supply is needed except the small

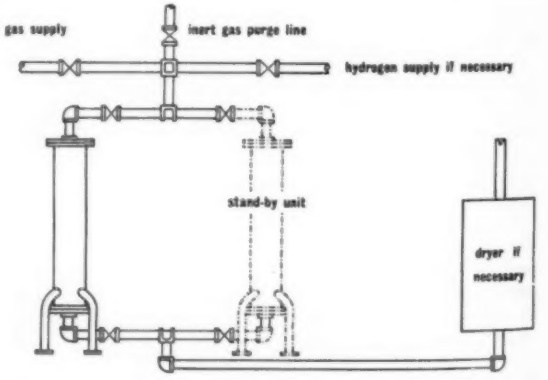

Diagram showing gas connections for Deoxo purifier required for all models over 200 cu. ft. per hour capacity.

amount needed by the regenerative dryer. The unit is connected to the gas supply line as shown in the illustrations and will operate indefinitely without attention, provided the installation instructions are followed.

It is proposed to offer 5–50, 200–50, 1,000–50, 2,500–50 and 5,000–50 models, the first figure in each case indicating the capacity in cu. ft. per hour. The maximum working pressure is 50 lb./sq. in. and the weight varies from 8 oz. to 490 lb. Larger models can be manufactured to meet customers requirements. The 5–50 model, which can be attached directly to the gas regulator of an ordinary hydrogen cylinder will doubtless have a particular appeal to those interested in pure hydrogen for experimental purposes.

Light Alloy Coach Bodies

FOUNDED in 1897, the firm of Thomas Harrington, Ltd., has seen many changes in coachbuilding since the days of the horse-drawn carriage. Towards the end of the nineteen twenties, it was realised by the directors that the larger motor-car manufacturers were gradually standardising types of car bodies and producing them themselves. The Company, therefore, gradually turned to the building of luxury passenger coachwork for commercial vehicles, and this remains its chief activity.

As chassis designs improved, the all-timber bodies gave place to a composite method of construction in which steel was used to give added support to the timber structure. Such a composite form of construction is still widely used to-day, but the all-metal steel body, with its longer working life, has gradually increased in popularity. The varying climates and conditions in which many passenger-carrying vehicles have to operate in different foreign countries have shown that in many cases the all-steel bodies do not fulfil the necessary requirements. Immediately prior to the War, the Company was considering the development of a body incorporating aluminium instead of steel in partial or complete form but the project had to be shelved.

Mainly in the light of experience gained in the design, development and manufacture of light-alloy aircraft components, a preliminary bus-body design was prepared towards the end of the War. This formed the basis of a considerable export business in light-alloy bus bodies and, in co-operation with the British Aluminium Co., Ltd., designs based on those which had been proved over a period of years were made available to interested parties overseas who, due to market conditions, could not order complete bodies from this country but who wished to improve their own forms of construction. In this way overseas coachbuilders were enabled to build a more advanced form of body in light-alloy, using materials manufactured and supplied by the British Aluminium Co., Ltd.

At first, light alloys were looked upon with some considerable doubt, but in actual fact it has now been proved that the material has many advantages over steel. With the changing design of chassis and increased running costs, the total weight of a vehicle to-day is more important than ever, and any saving which can be made, as for example by the use of light alloys, is most important. Furthermore, these materials are suitable for use in almost any climatic conditions where passenger vehicles are able to operate, as the metal is not affected by excessive humidity, nor can it be damaged by the various insects which attack timber overseas; corrosion resistance, too, is good.

Recently, as joint guests of Harringtons and the British Aluminium Company, a party of visitors inspected the new Harrington "Explorer" coach body.

The body is built of aluminium alloy extrusions and sheet metal of British Aluminium manufacture, and is held together with magnesium alloy rivets, "Pop" rivets, mild steel bolts and self-locking nuts. All materials are further protected by cadmium or chromium plating, and cellulose priming or paint where applicable, depending on the actual materials involved. Zinc chromate paste is used where dissimilar metals are in contact to prevent electrolytic corrosion.

The longitudinal and intermediate transverse members and miscellaneous secondary members in the floor are of extruded section and heavy gauge sheet metal riveted together, where possible, but bolted to facilitate assembly and replacement as necessary and suitably attached to the chassis. Three-quarter inch thick weather-proof resin-bonded plywood, Cuprinol treated, is fitted in sections and secured with metal screws.

The sides are constructed of "top hat" section pillars and cantrail channel section waist rail, angle section seat rail with heavy gauge truss panels from seat rail to floor structure. The structure is secured to the heavy side rail attached to the chassis outriggers. The interior is panelled with 18 S.W.G. and the exterior in 16 S.W.G. aluminium sheet.

The front is constructed of "top hat" pillars and roof "stick" with channel section waist rails, secondary members of extruded angle and sheet metal, panelled in 16 S.W.G. aluminium sheet.

The rear section is also constructed of "top hat" section pillars and roof "stick," with channel section waist rail and channel section cross bearers. It is panelled externally with 16 S.W.G. sheet and internally with 18 S.W.G. sheet.

The roof is fabricated in "top hat" section roof "sticks" with longitudinal rails of channel section and special channel section anchor rail for attachment of the roof unit to the cantrail. The roof is double skinned and suitably insulated between the two skins with Isoflex insulating material.

"Scientists to Industry"

FOUNDED in 1936, Nash & Thompson, Ltd. was responsible until 1945 for the design and development of the well-known Frazer-Nash gun-turret, and took a leading part in the intensive development of many types of radar scanner. In 1945 the Company assumed responsibility for research into problems associated with the design and development of a wide range of products for an important group of companies. Since that time, its activities have expanded continually. Now, the facilities and accumulated experience of the Company have been made available to industry in general, and a wide variety of industrial and scientific problems are being handled for leading organisations throughout the country. These include investigations into such matters as corrosion, electroplating, paint finishes, chemical and metallurgical analysis, colour matching and electronics.

We were recently privileged to visit the Nash and Thompson Laboratories and Workshops at Tolworth in Surrey, to see the facilities available and the type of work which is being undertaken. Briefly, the organisation comprises a staff of scientists, engineers and technicians, equipped with laboratories and workshops for carrying out investigations and for manufacturing specialised instruments and mechanisms.

The Laboratories and Workshops

The physics laboratory is fully equipped for investigations in both classical physics and modern electronic techniques, whilst the chemical and metallurgical laboratory can deal with chemical analysis, tensile and hardness testing, metallography, etc. On the engineering side there is a laboratory devoted to mechanical and general engineering and incorporating an experimental workshop which can undertake the production of prototypes of instruments. Other sections include the library, the design and drawing office, the machine and fitting shops and the pattern shop. The last named is engaged on the production of educational models, mock-ups, instrument cases and, of course, patterns for castings.

Interesting Instruments

During the last few years, a standard range of specialised scientific apparatus has been developed, and this is being continually augmented. The Company is also prepared to design and manufacture single instruments to meet special requirements and to produce such instruments in small batches.

Included in the range of instruments developed by the Company are a number of metallurgical interest. Reference has already been made in our columns to the metallurgical mounting press which incorporates a number of novel features. Based on a platinum resistance thermometer control element, and possessing a 2° C. differential between 0° C. and 1,000° C., the furnace controller is also of interest. Rather more unusual is the multipoint rapid temperature recorder, which scans a set of twenty thermocouples in six seconds and records the amplified voltages on a cathode ray oscillograph.

Three of the Nash & Thompson range of instruments are of interest in the application of paints to metals: the gloss-meter; the paint thickness meter; and the Holiday detector. The first-named has also been used as a means of assessing the surface finish of cold-finishing rolls, whilst the paint thickness meter is designed for the measurement of the thickness of paint films on non-ferrous metals. The Holiday detector is a rather specialised instrument designed for use on oil pipelines to detect the presence of minute pinholes in the protective coating of steel pipelines before the pipes are buried. A spring electrode encircling the pipe is maintained at a high electrical potential and moved along the pipe. When the electrode reaches a pinhole, a continuous spark arcs across to the pipe and warning is given by light flash and alarm bell. The spark also serves to burn out the hole, this rendering it more visible for filling.

One of the specialised pieces of apparatus has been designed for investigating the corrosive properties of the combustion products of gas. Eleven Mullite tubes are held at different temperatures ranging from 100° C to 1,000° C. The specimens under examination are placed in the tubes and over them are drawn the combustion products from a small gas flame at the mouth of each tube.

Space precludes a description of the whole range of instruments and equipment, but mention may be made of a gas flow-meter, a simple device for calibrating brittle lacquers used in stress analysis, and a cycling switch in which the on-off periods are independently variable from one second to 50 hours.

O.E.E.C. Countries to Restrict Use of Copper

JOINT action to alleviate the world shortage of copper, including common restrictions on the use of copper and copper alloys vital for defence purposes, has been agreed upon by the 18 Member countries of the Organisation for European Economic Co-operation.

As from October 1st, the use of copper and alloys with a copper content of 40% or more by weight is prohibited in the manufacture of more than two hundred different commodities ranging from window frames to cocktail shakers, and including building material, furniture, hardware and electrical appliances, transportation and refrigeration equipment, jewellery, gifts and novelties. The 18 European countries have also agreed not to export to each other commodities on the list containing copper in prohibited quantities, in order not to interfere with steps already taken to liberalise trade.

In certain cases—for example where articles containing copper are required for repairs or essential technical work—or where hardship is involved, Member countries may grant exemption from the new rules for their home markets. Exports to non-Member countries may continue in cases where the value of the commodities exported is high in relation to the value of their copper content, and general exemption for a period of three months may be granted to enable manufacturers and traders to dispose of their stocks of finished or semi-finished products on home or foreign markets. All Member countries are required to report to the O.E.E.C. by November 30th on the measures which they have taken to implement this decision, which marks a new step forward in European co-operation for the joint effort.

THE Board of the Steel Company of Wales, Ltd., announce that Mr. David J. Young, C.A., F.C.W.A., who has been Secretary of the Company since its formation in 1947, shall in future have the title of Secretary and Comptroller as this reflects more accurately the range of his responsibilities.

NEWS AND ANNOUNCEMENTS

European Metal Experts Leave for United States Study Tour

EIGHTY specialists, divided into three expert missions, have left for the United States under the O.E.E.C. Technical Assistance scheme. They will study respectively galvanising techniques, non-ferrous heavy metal fabrication, and smelting and refining of non-ferrous metals. On completion of a six weeks tour of research centres and industrial undertakings, the experts will return to Paris to draw up their reports for the 18 O.E.E.C. member countries.

These three missions bring up to 60 the number of technical assistance projects already carried into effect. Nearly a thousand European and American specialists have already taken part in this scheme to promote productivity in the industrial, agricultural and scientific fields by the exchange of technical knowledge and experience. In 25 cases, final reports have been prepared, many of which are now on sale to the public. A large number of further projects is under consideration by the O.E.E.C.

The United Kingdom is represented on two of the three missions :—

Galvanising Techniques : MR. R. W. BAILEY (Zinc Development Association), *Secretary to the Mission ;* MR. B. RYAN (Rylands Brothers, Ltd.) ; MR. R. STEWART (Smith & McLean, Ltd.) ; MR. A. CLIFT (Stewarts & Lloyds, Ltd.) ; MR. C. G. DAVIES (Richard Thomas & Baldwin, Ltd.).

Non-Ferrous Heavy Metal Fabrication : MR. C. F. DAVEY (T. Bolton & Sons, Ltd.) ; MR. H. H. SCHOLEFIELD (Telegraph Construction & Maintenance Co., Ltd.) ; MR. C. M. L. COWLEY (I.C.I., Ltd.) ; MR. E. N. LE PROVOST (Pirelli General Cable Works, Ltd.) ; MR. G. W. CHILD (Frederick Smith & Co.) ; MR. J. NORMAN (E. P. Jenks, Ltd.) ; MR. H. J. MILLER (British Insulated Callender's Cables, Ltd.).

The Institute of Metal Finishing

THE Council of the Electrodepositors' Technical Society announces that, in accordance with a Resolution passed at the last Annual General Meeting, application which was made some time ago for official incorporation has now been granted and as a result the Society is now an Incorporated Body. At the same time application was made for permission to adopt a new title embracing metal finishing generally (excluding only vitreous enamelling). This is in accordance with the general policy of the Society to extend its field beyond electrodeposition processes to include metal finishing generally with which the bulk of its members are vitally concerned. The new title by which the Society will henceforth be known is The Institute of Metal Finishing incorporating Electrodepositors' Technical Society Limited.

As a result of this change the original aims and objects of the Society—namely, to promote the study of, and disseminate information on, electrodeposition and cognate processes as widely as possible—are in no way affected and will, in fact, be pursued as vigorously as ever. The sphere of interest has now been significantly widened and the Council is of the opinion that this will bring with it corresponding benefit to the membership. The Executive Officers and Council of the new Institute remain unchanged. The present Executive Officers are Mr. H. Silman, B.Sc., F.R.I.C., A.M.I.Chem.E., F.I.M., President, Dr. S. Wernick, M.Sc., F.R.I.C., F.I.M., Honorary Secretary, and Mr. F. L. James, Honorary Treasurer.

1952 Machine Tool Exhibition

WE have received from the Machine Tool Trades' Association a copy of a tri-lingual leaflet (English-French-German) announcing the International Exhibition of Machine Tools to be staged at Olympia from September 17th to October 4th, 1952. This event is being organised by the Machine Tool Trades' Association of Great Britain, who have been responsible since World War II for the highly successful Machine Tool Exhibitions held in London, 1948, and Toronto, 1950 and 1951. As the latest machine tool and allied products of Great Britain, Europe and America will be installed and at work, an opportunity will be provided of inspecting the world's finest engineering production machinery and equipment. The announcement is made a year in advance of the event in order to provide visitors with ample time for making their arrangements to visit the Exhibition and securing requisite accommodation in London. Receipt of a pre-Exhibition Catalogue may be ensured by writing to the General Manager, Machine Tool Trades' Association, Victoria House, Southampton Row, London, W.C.1.

Sheet and Strip Conference

THE Annual Winter Conference of the Sheet and Strip Metal Users' Technical Association will be held on November 1st and 2nd at the Charing Cross Hotel. The proceedings will open with the Annual General Meeting, followed by the Official Opening of the Fourth Sheet Metal Working Exhibition. In the afternoon a paper on " Resistance Welding—Research Progress by the British Welding Research Association " will be presented by H. E. Dixon and H. G. Taylor. The technical sessions on November 2nd will comprise two discussions, in the morning : Tooling for Small Quantity Production ; and in the afternoon : Materials Handling for Presswork and Assembly Lines.

Foseco Conference

SOME fifty representatives attended the three-day Foseco conference held earlier this month by Foundry Services, Ltd. at the Welcombe Hotel, Stratford-upon-Avon. Apart from the Company's senior executives and 18 home representatives, agents and representatives from most European countries and from as far away as Australia, India and Canada were present. Home and overseas matters were considered in two separate and one joint session. Social activities included a visit to the Stratford Memorial Theatre, a visit to Oxford and the Morris Motors Works for the overseas representatives and their wives, and a farewell dinner on the Friday evening.

Sunvic Enter Pneumatic Control Field

SUNVIC CONTROLS, LTD. announce that they have made arrangements to manufacture and sell the range of pneumatic process control instruments manufactured by Moore Products Company, Philadelphia, U.S.A. These arrangements are exclusive throughout the British Commonwealth, with the exception of Canada, and non-exclusive throughout the rest of the world, except U.S.A. and Canada. The range of instruments will include pneumatic controllers, indicators, differential pressure transmitters, liquid level transmitters and controllers, thermometers, flow transmitters, valve positioners, manual control stations, relays, pilot valves, pressure regulators, etc. Enquiries and orders should be sent to Sunvic Controls, Ltd., Sunvic House, 10, Essex Street, Strand, London, W.C.2.

Simplified Productivity Report

LAST autumn, the Grey Ironfounding Productivity Team published a report on its visit to the United States, and the Council of Iron Foundry Associations sent a free copy to every iron foundry in the industry and encouraged employers to purchase extra copies for their key workers. To publicise still further the findings and conclusions of the team among the rank and file of the industry, the Council has now printed sufficient copies of a simply-written summary, entitled "What's going on over there," for each of the 150,000 workmen in the industry to receive a copy. These will be supplied, free of charge, to the 2,250 ironfounding employers.

Personal News

AT the Annual General Meeting of the Widnes Foundry & Engineering Co., Ltd. (one of the Thos. W. Ward Group of Companies) held recently, MR. RALPH CREDLAND, Assistant Managing Director of the Company for the past four years, was appointed Joint Managing Director.

DR. R. H. MYERS, who has recently been working at the Atomic Energy Research Establishment, Harwell, has been appointed the First Professor of Metallurgy at the University of Sydney.

DR. W. I. PUMPHREY, who has recently returned from a visit to the United States, as a Commonwealth Fellow, has been appointed Research Manager to Murex Welding Processes, Ltd., in succession to DR. E. C. ROLLASON, who took up his new appointment as Henry Bell Wortley Professor of Metallurgy, in the University of Liverpool, on October 1st. Dr. Pumphrey was leader of the welding research team of the Aluminium Development Association, at Birmingham University, from 1946 to 1949.

MR. G. H. FLETCHER has relinquished the position of General Manager of the Attercliffe Common works of the Metropolitan-Vickers Electrical Co., Ltd., on reaching retirement age.

MR. M. W. BARLOW has resigned from his position as Sales Manager of British Electro Metallurgical Co., Ltd., of Sheffield, to join Foundry Services, Ltd., as Manager of their newly established Ferro Alloy Division, and as such will be responsible for both the manufacture and sales of Foseco ferro alloy products.

DR. J. P. DENNISON, recently Assistant Lecturer in Metallurgy at the University of Leeds, has been appointed Lecturer in Metallurgy at University College, Swansea.

SIR CECIL WEIR, K.B.E., M.C., has joined the Board of Directors of The Pyrene Company, Ltd.

MR. C. H. DAVY has been appointed Works Director at the Renfrew and Dumbarton Works of Babcock & Wilcox, Ltd. Mr. Davy, who has been Chief Research Engineer of the Company since 1932, was appointed a Director in 1947. MR. I. M. LYON will continue in his present position as General Manager of Renfrew and Dumbarton Works, and MR. T. B. WEBB has been appointed Chief Research Engineer.

MR. S. M. LAWRENCE has been appointed Assistant Sales Manager, Unwrought and Special Products, of the British Aluminium Co., Ltd., in place of MR. R. M. WARRINGTON, who will be relinquishing his appointment with the Company on October 31st. MR. O. M. BRUCE PAYNE has taken over responsibility for the Technical Service Section of the Development Department, in place of Mr. Lawrence.

DR. N. E. RAMBUSH, Managing Director and Vice-Chairman of the Power-Gas Corporation, Ltd., has been elected Chairman of the Company following the retirement of MR. WILFRED BESWICK.

THE Metropolitan-Vickers Electrical Co., Ltd. has appointed MR. C. T. SCARF as Chief Engineer, Industrial Control Department. He succeeds MR. G. L. NEWMAN who is transferred to the staff of the Chief Electrical Engineer for special duties. MR. G. D. HARRADINE has been appointed Assistant Sales Manager, Industrial Control Department.

DR. R. GOODACRE has been appointed Secretary of the Leeds Metallurgical Society, in succession to MR. F. LINDARS.

MR. N. P. NEWMAN, Managing Director of Newman, Hender & Co., Ltd., has been elected Chairman of the British Valve Manufacturers' Association.

MR. C. G. GREEN has been appointed a Director of the Neepsend Steel & Tool Co., Ltd., in place of MR. A. HATTERSLEY, who has retired.

MR. J. MORGAN, who, since his resignation in August from the position as Chief Metallurgist with Magnal Products, Ltd., Bristol, has been at Foundry Services, Ltd., Birmingham, familiarising himself with Foseco products and the organisation, sailed recently, on the *Queen Elizabeth*, to take up his duties as Chief Metallurgist of Foundry Services (Canada), Ltd., Guelph, Ontario.

MR. H. A. MACCOLL, present Head of the Department of Metallurgy, has been appointed Principal of the County Technical College, Wednesbury, in succession to Mr. T. G. Bamford, who retires at the end of the year from the post which he has held for the past 23 years.

Obituary

THE British Driver-Harris Co., Ltd. of Manchester deeply regret to announce the death of Mr. S. C. Leoni, who was connected with the Driver-Harris Organisation for nearly 20 years, and was latterly Director of both the Italian and French Companies.

IT is with deep regret that we learn of the deaths of Mr. C. C. Howis and Mr. F. Walker in the recent train disaster at Weedon. Both men were engaged on important research work at the Kirkby Laboratories of British Insulated Callender's Cables, Ltd., Mr. Howis being the Company's Chemical Research Laboratory Manager there and Mr. Walker a research metallurgist.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Cold Cathode X-Ray Illuminator

Too often, in the past, the value of first class radiographs upon which the radiologist must base his report, has been nullified by indifferent facilities for viewing. To overcome this and to provide facilities at least equal to the standards required by the use of modern X-ray equipment, Watson & Sons (Electro-Medical) Ltd. have designed, in conjunction with the Illuminating Engineering Department of The General Electric Co. Ltd., a cold cathode X-ray illuminator. Cold cathode tubing has been chosen as the source of illumination because (1) it readily attains full intensity, (2) it does not generate heat, (3) there is no A.C. "flicker," (4) it can be dimmed without elaborate electrical gear, (5) it has a high luminous efficiency throughout its very long life.

The illuminator with the opal Perspex front cut away to show the specially designed cold cathode tube. The shape of this tube (in conjunction with the reflector) ensures the nearest approach to a homogeneous surface of light yet obtained.

The cold cathode tube used in the illuminator is internally coated with one of the new halophosphate powders. It is approximately 10 ft. in length and formed in the shape of a grid to obtain—in conjunction with a suitable reflector—an unusually high degree of distribution uniformity. The colour approximates closely that of daylight, and three degrees of illumination intensity are provided by a rotary switch with an additional "off" position. The intensity is sufficiently brilliant for the densest radiographs or for use when the general room lighting is exceptionally bright.

The housing of the illuminator has several features which have been introduced at the suggestion of leading radiologists. The illuminated surface extends almost to the full width, thereby facilitating the comparison of radiographs displayed on adjacent illuminators. The front is of opal Perspex which affords good diffusion and is stronger than glass, and a new type of spring clip for retaining radiographs extends the entire width. Radiographs of any size up to 14 by 17 in. are held firmly in position, and a useful feature is that two or more small radiographs can be held under the clip in any position along the top. A pair of brackets is provided for supporting developing hangers, and there is a detachable drip-tray of highly polished white plastic material. The illuminator is little wider than a 14 by 17 in. film and is thus ideally suited for use in groups of two or more. To facilitate this arrangement each illuminator is equipped with a socket on the inward-sloping part of the side, thus enabling several to be operated in parallel from a single electrical outlet. The shallow form of the illuminator will generally be found convenient because it occupies the minimum of desk space and enables the illuminator to be located on a narrow shelf. Finished in cream enamel, the illuminator has a current consumption of approximately 100 watts at 230 volts.

A flush mounted wall pattern is also available in which the illuminating system is identical with that described, but a chromium plated flange is supplied which lies flat against the wall, the housing itself being recessed. As the heat generated is inappreciable, no special ventilating arrangements are necessary. In cases where the wall is insufficiently thick to accommodate the 7½ in. thickness of the illuminator, it can be partly recessed and the space between the wall and the flange neatly filled with plaster sloped off on all four sides. Access to the cold cathode tube and electrical gear is gained from the front.

The General Electric Co., Ltd., Kingsway, London, W.C.2.

Colour Temperature Control of Butt Welding

A CONTINUOUS butt-welding process applied to chain links made a heavy demand on the attention of the operator, and to make the operation automatic and uniform a photo-electric equipment was supplied by Elcontrol, Ltd.

It was found that colour temperature of the weld was more reliable than the duration of the weld as a repetition standard, and to facilitate setting up the welding time, and as a safeguard, the photo-electric circuit was linked with an overriding timing circuit.

The equipment consists of two items: a photo-cell head, suitable for mounting within a few inches of the weld and housed in a cast aluminium case with tubular light shield and glass window, and the photo-electric amplifier and relay unit. The latter unit includes the photo-electric pyrometer circuit, having a sensitivity adjustment controlled by a knob on the panel, and the timing circuit. The 30-amp. primary contactor is included in the control unit. The timing circuit is adjustable and is set for a period slightly exceeding the weld time. The timing circuit controls the weld in the absence of a signal from the photo-cell. In normal operation, however, the correct weld-temperature will be reached before the end of the time period, and the contactor will be opened.

The circuits are interconnected in such a way that the welding current is switched off automatically in the event of a fault in any part of the equipment. The control unit includes a milliammeter indicating the rate of increase of photo-electric current as the weld temperature increases.

Elcontrol, Ltd., 10, *Wyndham Place, London, W.1.*

Imitation Nickel Plate Finish

In these days of difficulties regarding the use of nickel for electroplating purposes, an important advance in the science of metal colouring makes available to the trade the Niklit finish which gives attractive imitation white nickel finishes to brass and copper articles by simple immersion in a warm chemical bath. The process can be operated by unskilled labour and no complicated electrical equipment or solution analysis in production is required.

The Niklit finish is a change of surface and an integral part of the metal: it leaves no deposit, no dimensional change takes place, and fine limits can be maintained. As Niklit reproduces exactly the surface of the metal treated, there is no need for a further polishing operation after the treatment of a polished surface. If steel parts are first "coppered" or "brassed," they can be treated as copper or brass parts in Niklit. It is claimed that the finish is non-tarnishable and will not turn black, that there is no tedious cleaning of the article in use, and that quite apart from its good appearance it is a protection for the base metal.

Metal Processes, Ltd., Kingsbury Road, Erdington, Birmingham, 24.

Standard Polarographic Reagents

The main purpose of the new Polaritan range of reagents is to provide users of the polarograph with batch-tested materials specially suited to use as supporting electrolytes. The standards to which these chemicals are guaranteed to conform have been formulated by the Research Staff of the makers, Hopkin and Williams, Ltd., in collaboration with Tinsley (Industrial Instruments), Ltd. Limits have not been fixed for individual impurities but for each reagent a maximum permissible diffusion current has been specified. This provision limits the total of trace ions discharging over the prescribed voltage ranges, and these ranges and the conditions under which the tests are applied have been chosen to cover those circumstances in which the particular reagents are most commonly employed as polarographic base solutions. In certain circumstances it was impracticable to devise sufficiently comprehensive single tests, and in such cases the reagents are required to comply with two separate but complementary tests for polarographic purity. Where two reagents are themselves considered to be complementary (for example ammonium chloride and ammonia) suitable single tests employing both reagents have been formulated. The comprehensive limits set by these specifications will, it is thought, prove adequate for the majority of purposes and the issue, with each package of reagent, of a facsimile polarogram relating to the actual batch will provide the user with a valuable indication as to its adequacy for his particular purpose.

The present range includes ammonium chloride, ammonia solution (S.G. 0·880), barium chloride, citric acid, hydrochloric acid, nitric acid, potassium chloride, potassium hydroxide, potassium thiocyanate, sulphuric acid and tartaric acid. It is intended that this range shall be augmented, from time to time, by the addition of other substances commonly used as supporting electrolytes and, ultimately, to embrace the majority of such materials, together with certain ancillary reagents frequently used as maximum suppressors or as "pilot ions."

Hopkin and Williams, Ltd., Freshwater Road, Chadwell Heath, Essex.

Jenolite Chemical Sealer

Developed by Jenolite, Ltd., as a primer for application after Jenolizing, Jenolite Chemical Sealer is formulated to provide a safe system to render surfaces ready for a further application of paint, and to increase resistance to rust. Used in conjunction with Jenolite R.R.N. and A.K.S. many problems involved in the protection of metal have been overcome. Although Jenolizing prevents rust formation, the duration of the protection is ultimately dependent upon the finishing and the subsequent influences of atmospheric conditions. To safeguard iron and steel continually exposed to dampness and various degrees of temperature, a series of experiments was carried out using the Jenolite coating as the basis of the protective system.

During tests it was proved that the Chemical Sealer under development reacted with surplus Jenolite to provide an additional coat with superior corrosion-resisting qualities. In outdoor trials, the new Chemical Sealer was used immediately after Jenolizing heavily-rusted structural steelwork and rusty articles prior to finishing by ornamental top paint film. After loose rust had been removed and the surface Jenolized (sometimes using a wire brush for agitation) the now dissolved rust was wiped away and, where necessary, a second coat of Jenolite was thinly brushed on. This was allowed to air dry for 20 minutes, after which the metal surface was still found to be both moist and tacky. Over this, a coat of Chemical Sealer was applied by brush and pistol, and allowed to stand for 24 hours prior to final painting. The Chemical Sealer "touch dries" in 20 minutes and "dries hard" in 6 hours, but as the test was carried out on structural steelwork *in situ* on a wet day, the 24 hour drying period was taken as a precaution. Chemical Sealer can, of course, be oven-dried when, for instance, 20/30 minutes at 250° F. will suffice. This proved the most satisfactory system, the physical quality of the Chemical Sealer being such that it is absorbed by the Jenolite coating, also mixing with Jenolite surplus, thus achieving perfect paint adhesion. Any type of finishing paint can be employed (except cellulose), pigment necessarily varying according to the job. Chemical Sealer is supplied in red, grey, black and green and this, acting as an undercoat, gives added hue to the finishing colour. It can also be used subsequent to keying aluminium by Jenolite A.K.S., and where aluminium is used in conjunction with copper or lead, Chemical Sealer applied to the junction inhibits the electrochemical action normally set up.

Jenolite, Ltd., 43, *Piazza Chambers, Covent Garden, London, W.C.2.*

CURRENT LITERATURE

Book Notices

GUIDE TO FOREIGN LITERATURE ON METALS

Published by The Richard Rimbach Associates, 921, Ridge Avenue, Pittsburgh 21, Pa. Price $2.50.

In this excellent guide to foreign sources of metallurgical literature, which was prepared by John T. Milek, there are nine sections dealing with associations, periodicals, abstract services, standardising organisations, statistical sources, directories of industries, metallurgical books, bibliographies, and metallurgical abbreviations. Among some of the subjects covered in the section on foreign books (in print from 1946 to 1949) are alloys, aluminium, bearing metals, chemical analysis, copper, corrosion, economics, foundry and smelting, general metallurgy, light metals and numerous other metallurgical group headings. One section also lists official sources of metal industry statistics. This collection of references on foreign information relating to metals should prove to be most useful to engineers, statisticians, metallurgists, sales and advertising personnel, librarians, and others who may seek access to information on foreign literature in metallurgical and related fields.

FIFTY YEARS OF BRITISH STANDARDS 1901–1951

The British Standards Institution, Sales Department, 24, Victoria Street, London, S.W.1. 10s. 6d. post free.

This specially-written history is an illuminating record of the British Standards movement—showing how it originated in 1901 as the Engineering Standards Committee, how it has developed and contributed increasingly to the national economy, and how the British model, in this as in many other fields, has been followed throughout the civilised world.

Special chapters deal with the influence of the standards movement in the two world wars and with its spread to all the countries of the British Commonwealth; with the relationship between standardisation and research; and with some of the brilliant figures in industry, science and the arts who have helped to build the movement during its half century of steady growth.

Though the subject is highly specialised, the book is written in an easy, readable style, and it throws a long-needed light upon a facet of British industrial genius about which too many people know far too little.

BRITISH STANDARDS 1951 YEAR BOOK

The British Standards Institution, Sales Department, 24, Victoria Street, London, S.W.1. 7s. 6d. post free.

The new edition of this invaluable reference work has just been published. Well over half of its 400 pages are devoted to a list of the 1,700 British Standards current at 31st December, 1950, with a brief description of the subject-matter and scope of each one. A complete subject index simplifies reference. In addition, a supplement listing the British Standards issued between 1st January and 31st March, 1951, is included. The year book also lists the current membership of the General Council, which guides the Institution's general policy; of the four Divisional Councils (Building, Chemical, Engineering and Textile), which direct the standardisation work; and of the 56 Industry Standards Committees, each representing a major British industry and through which that work is delegated to some 2,000 Technical Committees and Sub-committees, whose members bring specialist technical and commercial knowledge to bear upon every standardisation project dealt with.

A GUIDE TO THE PROPER TREATMENT OF FACTORY VISITORS

"Most companies spend large sums on advertising, one of their objects being to remind the public of the service which can be expected from the factory. It seems absurd not to take trouble with members of the public who actually cross the threshold, but too often the ordinary visitor has to wait five or ten minutes before anybody takes any notice of him, and this is just a foretaste of the treatment he will receive throughout his visit." With a view to remedying this state of affairs the Training Department, TI (Group Services), Ltd., has circulated to all concerned a new training booklet entitled "A Guide to the Proper Treatment of Factory Visitors," from which the foregoing statement was extracted.

While a light approach has been adopted, the subject is, nevertheless, considered to be one of great importance, particularly where overseas customers are concerned, and in this connection the reader is advised to consider the type and standard of entertainment usual in the visitor's own country. Those with experience as visitors or visitees will readily appreciate that the light and entertaining manner in which the booklet is written serves to emphasize, rather than hide, the sound common sense which prevails throughout. Permission has been granted the British Association for Commercial and Industrial Education, of Management House, Hill Street, London, W.1., to publish a special edition for general sale.

Trade Publications

The results of more than 25 years of laboratory and field study by the technical staff of the company on the nature of corrosive attack on copper and copper alloys have been published by the American Brass Company. This publication, believed to be the first of its type in the industry, explains the chemical and physical nature of corrosion in its various forms. Among the subjects discussed are theory of corrosion, discussion of corrosion resistance, rating charts, typical industrial uses of copper alloys, composition, mechanical and physical properties of alloys, welding rods and a questionnaire on process conditions. The booklet is well illustrated with significant photomicrographs. The tabulation indicating the relative corrosion resistance of the principle types of copper and alloys when in contact with 183 different corroding agents should prove most valuable to material engineers in selecting copper alloys best suited for a

specified use. This 24-page booklet entitled "Corrosion Resistance of Copper and Copper Alloys (Anaconda Publication B36)" may be obtained from the American Brass Company, Waterbury 20, Conn.

ALTHOUGH conservation of alloying elements has become a vital necessity since the preparation of "The Mechanical Properties of Nickel Alloy Steels" by the Mond Nickel Company, it is hoped that the publication will serve a useful purpose as it includes some "economy" steels and provides data on standard nickel alloy steels as a basis of comparison with other "economy" steels which may be developed later. Moreover, many of the standard steels will again be available when the present emergency is passed. Properties are presented in tabular form whilst the effect of variation in tempering temperature is graphically displayed. The production is of the high standard one has come to associate with Mond Nickel publications.

IN highly technical, yet clearly defined language, Reynolds Metal Company has published a booklet on "Aluminium Structural Design." Amply illustrated with photographs, drawings and tables, the three main sections are devoted to the mechanical properties of aluminium alloys, structural design considerations and fabricating considerations. Copies may be obtained on request (on a Company letter-head) to Export Division, Reynolds Metal Company, 19, East 47th Street, New York, 17, N.Y.

In a handsome, illustrated booklet entitled "Post War Report," Gibbons Bros., Ltd., Dudley, tells the story of its activities since the cessation of hostilities in 1945. In spite of material and labour shortages, the Company has been able to carry out work of a more varied type than in any preceding five years of its history, and a glance through its pages, in which typical examples are illustrated, emphasises this point. Gas works' plant, furnace and handling plant, pottery ovens and kilns, gas producers and coke ovens, all find a place, whilst the booklet is completed by a series of illustrations showing the post-war expansion of Gibbons' own works.

The Quasi Arc Co., Ltd., have produced a complete catalogue, listing and illustrating the entire range of electrodes, plants and accessories which they manufacture for automatic and manual arc welding of ferrous and non-ferrous materials. The Unionmelt process for submerged melt automatic arc welding is described, together with details of the range of Unionmelt plant now available in this country. Copies may be obtained, free of charge, from the Quasi Arc Co., Ltd., Bilston, Staffs.

The series of alloys formed by nickel and iron is remarkable for its wide range of magnetic and thermal characteristics. To supplement the full description of magnetic characteristics contained in an earlier publication, the Mond Nickel Company has recently issued a comprehensive summary of those associated with thermal changes, under the title "Physical Properties of the Nickel-Iron Alloys." This publication, obtainable free of charge from the Company at Sunderland House, Curzon Street, London, W.1, gives data on the tensile strength, hardness and electrical resistance of the binary alloys and the elastic and expansion properties of both the binary and the more complex alloys. The section dealing with applications describes the use of these alloys for a variety of temperature control devices, for expansion control in engineering components and for glass-metal seals.

A new publication issued by Henry Wiggin & Co., Ltd., presents in a convenient form all the available information on the Nimonic series of alloys, including some data only recently determined as a result of the long-time creep testing which is, of course, still in progress. The list of typical applications shows that these nickel-chromium alloys, in addition to their use for gas turbine blades, have a wide field of application for many other components operating at elevated temperatures. Copies are available, free of charge, from Messrs. Henry Wiggin & Co., Ltd., Wiggin Street, Birmingham, 16.

Designed to combat hot spots in industry, details of "Tornado" man-cooling fans are given in a revised and up-to-date brochure issued by Keith Blackman, Ltd., Mill Mead Road, Tottenham, London, N.17. These fans have been marketed for well over 10 years and have found application in a wide variety of industries, including iron and steel works, foundries, etc.

To commemorate the termination of the Festival of Britain, Almin, Ltd. have produced an attractive souvenir brochure devoted to the Dome of Discovery—"the most outstanding structure on the South Bank." The photographs illustrate the manufacture of several structural items, together with the various stages of erection on site.

Books Received

"Electroplating and the Engineer," by Alan Whittaker. "Mechanical World" Monographs—64. 87 pp. inc. Tables. Manchester, 1951. Emmott & Co., Ltd. 4s. net.

"Powder Metallurgy." Volume 9 of Selected Government Research Reports. 159 pp. London, 1951. Issued by the Ministry of Supply. Published for the Technical Information and Documents Unit of the Department of Scientific and Industrial Research by H.M. Stationery Office. 18s. net.

"Die Metallwirtschaft der Schweiz und ihre Nebengeniete." 120 pp. Zurich, 1951. Verlag für Wirtschaftsliteratur G.m.b.H. Also obtainable from H. K. Lewis & Co., Ltd., London. 21s.

"Electro-Plating." A Survey of Modern Practice Including the Analysis of Solutions. 6th Edn. By Samuel Field, A.R.C.Sc., and A. Dudley Weill. 546 pp. inc. index. London, 1951. Sir Isaac Pitman & Sons, Ltd. 20s. net.

"Magnetic and Electrical Methods of Non-Destructive Testing." By D. M. Lewis, B.Sc., A.I.M. Foreword by Professor L. F. Bates, Ph.D., D.Sc., F.R.S. Report prepared for the Magnetic and Electrical Methods Sub-Committee of the British Iron and Steel Research Association. 242 pp. inc. index. London, 1951. George Allen & Unwin, Ltd. 35s. net.

"Wavelength Tables of Sensitive Lines," by L. H. Ahrens, D.Sc., F.R.I.C. 86 pp. Cambridge, Mass., 1951. Addison-Wesley Press, Inc. $3.00.

METALLURGICAL DIGEST

Centrifugal Casting of Pipe in Sand-Lined Mould

By D. R. Daniel

THERE is no definite knowledge as to when and where the first iron pipe was made. It may be assumed, however, that it followed closely upon the development of, or was produced coincidently with, the cannon, the earliest mention of which is found in the year book of the City of Ghent, dated 1313. An old German catalogue of a pipe manufacturer relates that cast-iron pipe was used in the water system of Dillenberg, Germany, in 1455. Cast-iron pipe laid at Longensalya, Germany, in 1662, is still in use to-day—a service record of 285 years. In 1661 cast-iron pipe was installed for water service at Braunfels, Germany. When replaced in 1932 with larger pipe, the original pipe, after 271 years, was in excellent condition.

Authentic records of cast-iron underground water-conduit installations reveal that several lines of various diameters were laid, by order of Louis XIV, near Paris, France. These were to carry water from the reservoirs of Picardy, to those of Montbauron and, together with the spring-water conduits, to supply the town and parks of Versailles. In all, a length of about 26,000 ft. was manufactured and laid between 1664 and 1688, or about 275 years ago. All these pipes consist of 3 ft. 3 in. lengths, joined by means of bolted flanges. They are of considerable weight and still serve their purpose satisfactorily. The few repairs which have been required have generally been necessitated by the bad condition of the flange bolts which have rusted out. The pipes were cast horizontally. The flange joints were made with a thick lead gasket, sometimes as much as $\frac{7}{8}$ in. thick. The system is under considerable pressure.

As early as 1744 the Chelsea Water Company, London, laid a 12 in. flanged cast iron pipe which was relaid in 1791 because of the joints "being perished." It was the engineer of that water company, Thomas Simpson, who designed the first bell and spigot pipe. This was about 1785, at which time an experimental section of pipe was laid with lead and jute joints, which proved so successful that the entire system of all the London Companies was gradually relaid with bell and spigot cast-iron pipe, the uninterrupted service record of which now covers 135 to 160 years. This bell and spigot design of joint is still the accepted standard for most cast-iron water mains.

In 1804 the Watering Committee of Philadelphia decided to import from England a small quantity of cast-iron pipe to be laid as an experiment. This was bell and spigot pipe in 9 ft. lengths. It is believed that this was the first cast-iron pipe laid in the United States.

As far as is known the first cast-iron pipe was moulded in sand, using wooden boxes or flasks, and cast in a horizontal position. This was the only method of casting pipe for over 300 years, and is still used for casting short lengths. As the standard length increased from about 3 ft. to 6 ft., then to 9 ft. and later to 12 ft. and 16 ft., the method of casting changed from the horizontal to a sloping position, and finally to a vertical position. The pit-cast vertical method of production was developed by the Links Foundry in 1846 at Montrose, Scotland. This method generally replaced the horizontal method for all pipe longer than 9 ft.

The change to the vertical method was made in order to secure a more uniform thickness of wall and better distribution of metal, as well as to ensure cleaner and stronger castings by bringing all dirt or slag to the top of the mould into a large runner which is subsequently broken or cut off. A sectional view of a vertical mould is shown in Fig. 1. This method, which came into use in 1834, was universally applied until the early 1920's and is still in use for the casting of large-diameter pipe.

Probably the most outstanding development in pipe manufacture took place about 1922. Two new centrifugal processes marked a radical departure from the established methods of horizontal and vertical pit casting—the metal mould process and the sand-lined mould process. In 1922 the author's company—American Cast Iron Pipe Company—started the development of the process of centrifugal casting of pipe in sand-lined moulds, and during the three years that followed the process was perfected. In 1923 a small production unit, consisting of four machines making a 12-ft. pipe in 4 and 6 in. sizes, was completed and operated through 1925. In 1926 the first centrifugal casting shop was built, consisting of 12 machines for making pipe in 3 through 12 in. sizes in 16 ft. lengths. This shop, with 25 years of constant improvement, is the present-day pipe-producing unit, 3 through 12 in. In 1930 the second centrifugal shop was built for casting sizes 14 through 24 in., which was later modified to include 14 through 48 in., all in 16 ft. lengths.

The moulding boxes are machined on all points to make possible rapid

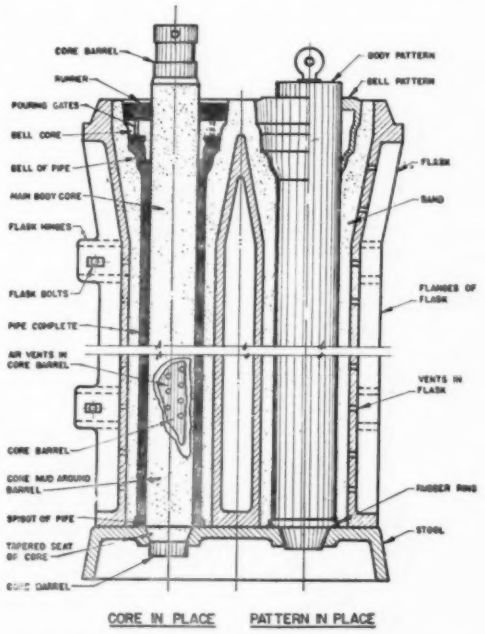

Fig. 1.—Sectional view of a pit-cast mould.

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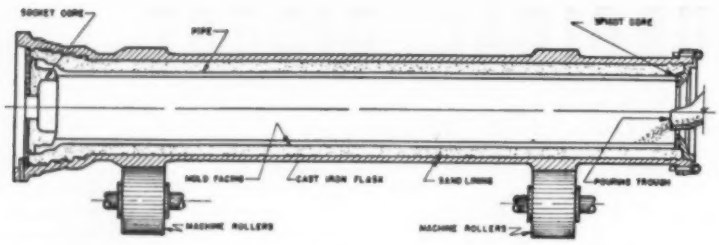


Fig. 2.—Sectional view of a centrifugally cast pipe in a sand-lined mould.

duplicate work and each box is designed for one pipe. There are two 3-way ramming stations in the centrifugal casting shop. Empty moulding boxes come down a run and are received on a tilting table in the run which delivers them into the vertical position on the ramming stations. The moulding boxes are handled three at a time and the two ramming stations are used to alternate the work so that ramming can proceed on one station while on the other station moulds are completed and the box change made. When the moulding boxes are in the vertical position and properly located on the ramming stools, the patterns are located. The ramming is accomplished by means of special pneumatic rammers running at the rate of 600 strokes per minute and using an air pressure of 100 lb./sq. in. The ramming time depends upon the size of the mould and the size of the sand stream. It varies from 1 min. 10 sec. to 1 min. 30 sec. Each moulding box is revolved at a speed that will allow a layer of sand approximately 3½ in. thick for each revolution of the mould. The thickness of sand between pattern and moulding box is approximately 2 in. for all sizes.

The patterns are drawn from their moulds three at a time and the moulds are lifted off the ramming stools and a facing applied while they are in a vertical position. The moulds are then returned to a horizontal position by a tilting table and are then ready to move forward on a skin drying run to the casting bay. Skin-drying to a depth of ¼ to ⅜ in. is done by gas torches firing into each end alternately and occupies about 4 minutes. Just as the skin drying operation is finished the spigot core is placed in position and secured by a bolted plate.

The mould is located in the casting machine and is rotated at a speed of about 40 r.p.m. while the correct amount of iron is poured, and as soon as casting is completed the speed of rotation is accelerated to as high as 1,700 r.p.m. for a light 3-in. pipe to 336 r.p.m. for a heavy 48 in. pipe. The machine rollers which rotate the moulds are shown in Fig. 2.

The moulding box with the sand and pipe in it passes to a stripping machine which is built on a 37° incline and is capable of dealing with three moulding boxes at a time. A revolving tube with a special cutter is advanced into the moulding box at the rate 20 ft./min. cutting the sand between the pipe and the moulding box. The pipe is then removed from the mould and transferred to the cleaning department. It is noteworthy that the inside is cleaned by grinders entering each end.

The Cracking of an Al-Mg Alloy in Contact with Mercury

By P. A. Jacquet and A. R. Weill

THE investigation, which is described in detail, was carried out on specimens from compressed air cylinders of Al–7% Mg. alloy, a number of which had burst in service during which they were exposed to sea-air on board ship. The mercurous nitrate test as applied to brass was used in an attempt to discover the cause of this bursting and structural heterogeneities. The test solution used contained 100 g. of mercurous nitrate and 16 ml. of concentrated nitric acid per litre. The specimens to be tested were first subjected to a short period of electrolytic polishing in an acetic anhydride-perchloric acid solution, washing and drying immediately prior to immersion in the test solution. Specimens were examined for cracking every three minutes. The mercurous nitrate tests were supplemented by micrographic and X-ray examinations and investigations of the effects of working and annealing.

Cracking of the Al.-Mg. specimens in the mercurous nitrate test was found to be related to differences in orientation of the grains over the wall-thickness of the cylinders which had been hot and cold formed without a final anneal, these differences in orientation being associated with internal stresses. Cracking was intercrystalline but differed from stress corrosion in that it was not dependent on the presence of a precipitated phase in the grain boundaries.

The tendency to crack in the mercurous nitrate test was eliminated by annealing at 160° C., the elimination of the tendency to crack occurring before the X-ray diffraction diagrams showed any substantial change.

The mercurous nitrate test can serve as a means of detecting the presence of internal stresses which may bring about stress corrosion should an aluminium-magnesium alloy be otherwise rendered susceptible to it.

From *Revue de l'Aluminium*, **27**, 442-451, December 1950, and **28**, 4-10, January 1951. (Abstracted in *Light Metals Bulletin*, **13**,9,308-309.)

Removing Surface Defects on Castings

SAND encrustations, fins, pads, chaplets or chill nails, and other forms of excess metal are said to be removed from castings quickly, easily and economically by means of an oxyacetylene method developed by Linde Air Products Co.

The new process, called Powder-Washing, utilises a special blowpipe, equipped with an external powder-washing attachment. With this apparatus, an iron-rich powder is fed through oxyacetylene preheat flames into a low-velocity oxygen stream where it burns and produces superheated liquid iron oxide. Heat from the combustion of the powder, and from the slag, simplifies and speeds the removal of metal and metal-sand mixtures. Wherever the powder-fed flame is directed against a casting, the metal surface is brought quickly to temperature, and then it is oxidized when blown away by the oxygen stream. After Powder-Washing, the surfaces of castings are left smooth, clean and close to tolerance. There is no undercutting, and no torn metal.

From *The Iron Age*, July 19th, 1951, p. 101.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS

OCTOBER, 1951 Vol. XLIV, No. 264

The Absorptiometric Determination of Magnesium

By A. Bacon

Metallurgy Department, Royal Aircraft Establishment, Farnborough.

This article describes an investigation into the use of Titan Yellow and Solochrome Cyanine R.S. as reagents for the absorptiometric determination of magnesium. A method is described for the rapid determination of magnesium in aluminium alloys using Solochrome Cyanine R.S.

THE absorptiometric determination of several of the alloying metals in aluminium alloys has been previously described and the colour reactions employed fully discussed.[1] The time required for the complete analysis of aluminium alloys would be considerably curtailed by the introduction of a rapid absorptiometric method for magnesium, and as it was considered that Solochrome Cyanine R.S. might prove more convenient than the Titan Yellow employed by Stross[2] the properties of both reagents were studied.

A tungsten filament lamp was used as the light source for all experiments, and the transmission curves given were obtained by plotting the relative transmissions against the wavelengths of maximum transmission of the filters.

Titan Yellow Reagent

Relative Absorptions.—The transmission curve for alkaline solutions of Titan Yellow is given in Fig. 1 and shows maximum absorption to occur in the violet. The introduction of magnesium into such solutions results in a marked increase of absorption over the blue-green to yellow wave band. For the measurement of the absorption due to the magnesium compound, there is little to choose between green or yellow-green filters, but the absorption due to Titan Yellow is considerably less when the latter are used.

Effect of pH.—In order to study the effect of the hydrogen ion concentration on the development of the colour due to magnesium, a series of tests was conducted at constant concentrations of magnesium (0·005 g./litre), sodium sulphate (0·005N) and Titan Yellow (0·10 g./litre). The volume was maintained constant at 100 ml. and caustic soda additions were made to vary the *p*H over the range 10·0–13·0. The approximate *p*H was calculated from the caustic soda concentration by the following equation :

$$pH = 14{\cdot}0 - \log \frac{1}{c}$$

where c is the caustic soda concentration in terms of normality. In the region of 11·0 the *p*H was measured by means of a glass electrode and agreed closely with the value obtained by calculation. The absorption of yellow-green light by the coloured solutions was measured on the Spekker, within 5 minutes, using 4 cm. cells. The results are plotted in Fig. 2, and it can be seen

1 Davis, H.C., and Bacon, A. A study of some chemical reactions employed in photometric analysis. *J. Soc. Chem.*, 1948, 67.
2 Stross, W. The rapid photometric determination of magnesium in aluminium alloys. *Analyst*, 1942, 67,317.

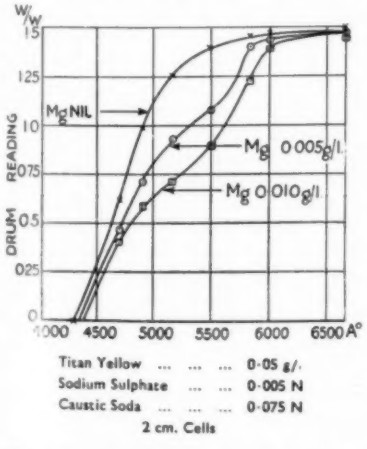


Fig. 1.—Transmission Curve for magnesium (Titan Yellow).

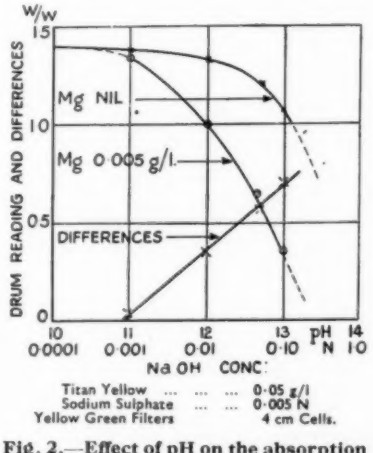


Fig. 2.—Effect of pH on the absorption by the reagent and magnesium complex.

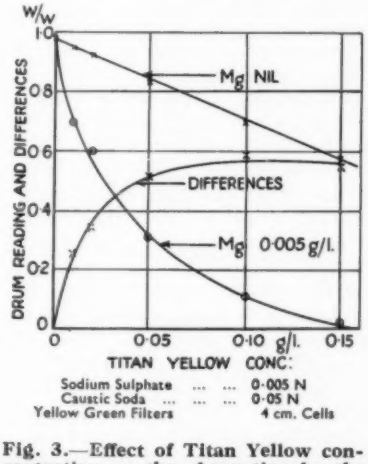


Fig. 3.—Effect of Titan Yellow concentration on the absorption by the reagent and magnesium complex.

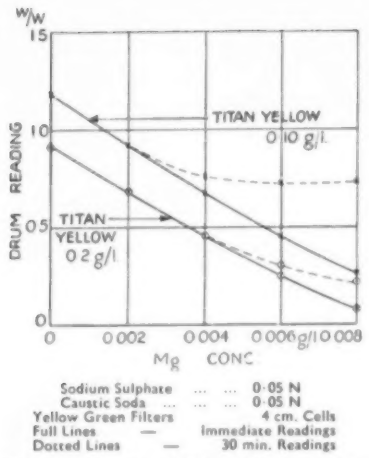


Fig. 4.—Effect of Titan Yellow concentration on the stability of the magnesium complex.

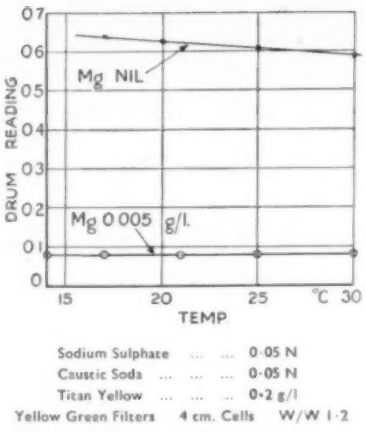


Fig. 5.—Effect of temperature on the absorption by the reagent and magnesium complex.

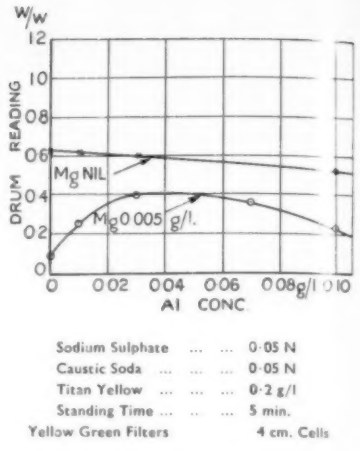


Fig. 6.—Effect of aluminium on the absorption by the reagent and magnesium complex.

that the absorption by Titan Yellow increases rapidly above *p*H 12·0, whereas in the presence of magnesium the absorption commences to increase rapidly on exceeding *p*H 11·0.

By plotting the differences in absorption against *p*H, it can be seen that the sensitivity is negligible at *p*H 11·0, but increases with rise in *p*H above this value. The maximum *p*H which can be employed is limited by the increasing absorption by the Titan Yellow.

Titan Yellow/Magnesium Ratio.—A series of tests was conducted at constant concentrations of magnesium (0·005 g./litre) and caustic soda (0·05N). A sodium sulphate concentration of 0·005N was employed since it was found that, when the sodium sulphate concentration was increased to 0·25N, the solutions containing 0·005 g./litre magnesium became turbid. The concentration of Titan Yellow was varied between nil and 0·15 g. litre. The absorption of yellow-green light by the coloured solutions was measured within 5 minutes, on the Spekker, using 4 cm. cells. The results are given in Fig. 3, and show that, in the absence of magnesium, the absorption progressively increases with reagent concentration and that 0·005 g./litre magnesium requires about 0·10 g./litre reagent concentration to produce maximum colour.

It can be seen from Fig. 4, however, that 0·10 g. litre Titan Yellow is insufficient to stabilise the colour at this concentration of magnesium. This graph was constructed from a similar series of tests conducted at two reagent concentrations (0·10 and 0·2 g./litre), the magnesium being varied over the range 0–0·008 g./litre.

The Titan Yellow reagent was added as an 0·1% aqueous solution before making the solutions alkaline.

Effect of Temperature.—It can be seen from Fig. 5 that the temperature has little effect on the absorption by Titan Yellow solutions and that when magnesium is present the temperature effect is negligible.

Effect of Aluminium.—The results of experiments conducted to determine the effect of aluminium on the Titan Yellow reaction are shown in Fig. 6, and it can be seen that although, in the absence of magnesium, aluminium has little influence on the absorption by the Titan Yellow, in the presence of magnesium, traces of aluminium have a serious effect.

Additions of sodium citrate necessitated an increasingly higher *p*H for maximum colour formation and resulted in some magnesium being undetected, the amount increasing with the citrate concentration. Above this limit of detection, the sensitivity remained unaffected by citrate additions. The interference due to aluminium shown in Fig. 6, was not removed by sodium citrate. It is essential, therefore, that the solution should be freed from aluminium before applying the Titan Yellow reaction.

Solochrome Cyanine R.S. Reagent

This reagent has previously been used for the absorptiometric determination of aluminium; it changes from yellow to red over the *p*H range 10·0 to 12·0 and is more sensitive to *p*H changes than Titan Yellow.

Relative Absorptions.—The transmission curve for alkaline solutions of Solochrome Cyanine R.S. is given in Fig. 7 and shows maximum absorption to occur in the violet and blue wave bands. The introduction of magnesium into such solutions results in a marked increase of absorption in the yellow-green wave band.

Effect of pH.—In order to study the effect of the hydrogen ion concentration on the development of colour due to magnesium, a series of tests was conducted at constant concentrations of magnesium (0·010 g./litre), sodium sulphate (0·01N) and Solochrome Cyanine R.S. (0·10 g./litre). The volume was maintained constant at 100 ml. and caustic soda additions were made to vary the *p*H over the range 9·0 to 13·0. As in the case of the Titan Yellow reagent, the *p*H was calculated from the caustic soda concentration, and values below *p*H 11·0 were measured by means of a glass electrode. The absorptions were measured on the Spekker, within 5 minutes of developing the colour. A similar series was conducted omitting the magnesium addition. The results are plotted in Fig. 8 and show that the absorption by the Solochrome Cyanine R.S. increases rapidly above *p*H 11·0 whereas, in the presence of magnesium, the absorption commences to increase rapidly on exceeding *p*H 9·5. The difference in absorption, unlike Titan Yellow, reaches a maximum at *p*H 11·0 and falls again above this *p*H.

Influence of Sodium Salts.—It can be seen by comparing Figs. 8 and 9, that an increase in the sodium

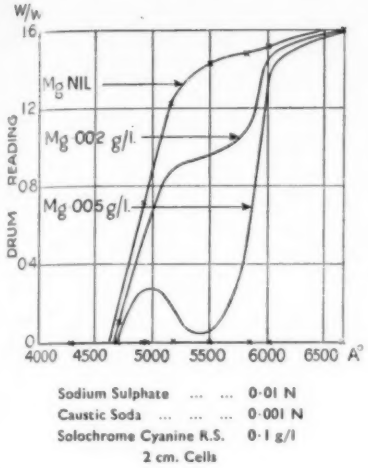


Fig. 7.—Transmission curve for magnesium (Solochrome Cyanine R.S.).

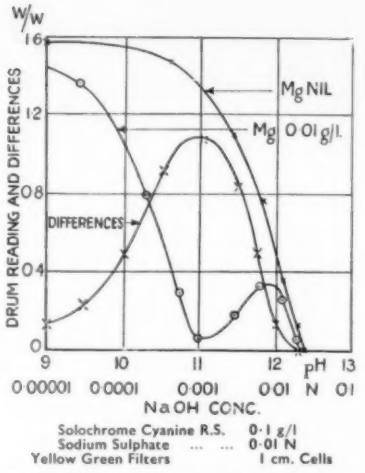


Fig. 8.—Effect of pH on the absorption by the reagent and magnesium complex (sodium sulphate 0·01N).

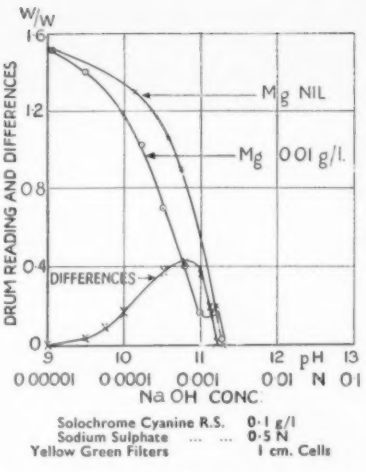


Fig. 9.—Effect of pH on the absorption by the reagent and magnesium complex (sodium sulphate 0·5N).

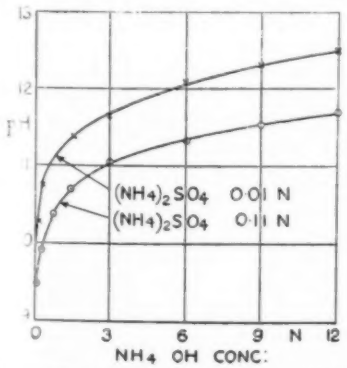
sulphate concentration increases the absorption by solutions not containing magnesium whilst those containing magnesium are unaffected, so that the difference in absorption is considerably decreased by increasing the sodium sulphate concentration. The maximum difference still occurs at *p*H 11·0.

Control of pH.—To control the *p*H between 10·5 and 11·5 it can be calculated that the caustic soda concentration should not be higher than 0·0032N nor lower than 0·00032N. Close control of the *p*H in the region of 11·0 by caustic soda is therefore not practicable.

For a similar *p*H range, using ammonia, the concentration can vary between 0·2 and 2 N at an ammonium sulphate concentration of 0·01N. At an ammonium sulphate concentration of 0·11N the ammonia concentration can vary between 1 and 8 N for the *p*H range 10·5 to 11·5. This is shown in Fig. 10.

Effect of pH (Ammonia Solutions).—A series of tests was conducted at constant concentrations of magnesium (0·010 g./litre), ammonium sulphate (0·01N) and Solochrome Cyanine R.S. (0·10 g./litre). The *p*H was varied by ammonia additions and measured using a glass electrode, neutralising of the acid magnesium sulphate addition resulting in the ammonium sulphate content stated. A similar series was conducted omitting the magnesium addition and substituting a sulphuric acid addition to produce the ammonium sulphate. The absorptions were measured on the Spekker within 5 minutes of developing the colour.

It can be seen from a plot of the results (Fig. 11), that the absorption by the Solochrome Cyanine R.S. increases up to *p*H 11·0 but differs from the behaviour in caustic soda solutions by decreasing above this *p*H. In the presence of magnesium, the relation between absorption and *p*H is very similar to that obtained in caustic soda solutions. Further experiments using an ammonium sulphate concentration of 0·11N showed the absorption due to magnesium to be critically dependent on the ammonium sulphate concentration (Fig. 11). The



10.—pH curve for ammonia solutions.

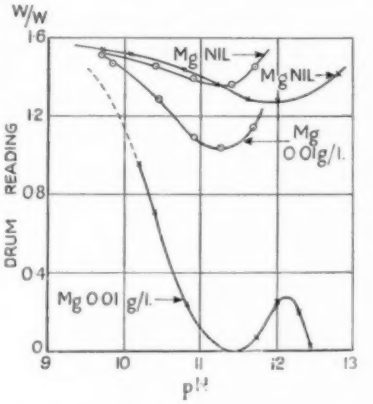


Fig. 11.—Effect of pH on the absorption by the reagent and magnesium complex (ammonia solutions).

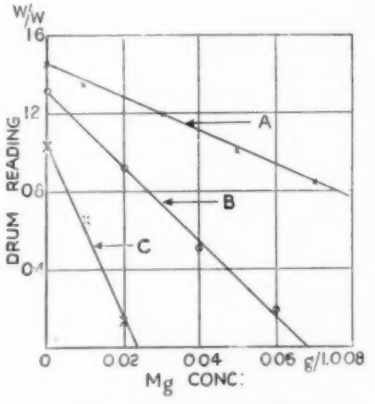


Fig. 12.—Effect of Solochrome Cyanine R.S. concentration on the sensitivity.

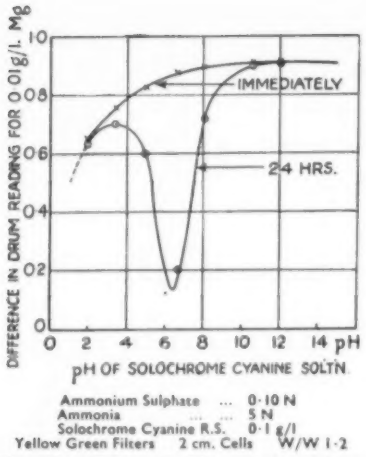

DIFFERENCE IN DRUM READING FOR 0·01 g/l. Mg
IMMEDIATELY
24 HRS.
pH OF SOLOCHROME CYANINE SOLTN
Ammonium Sulphate ... 0·10 N
Ammonia ... ... 5 N
Solochrome Cyanine R.S. 0·1 g/l
Yellow Green Filters 2 cm. Cells W/W 1·2

Fig. 13.—Effect of pH on the stability of the Solochrome Cyanine R.S. reagent.

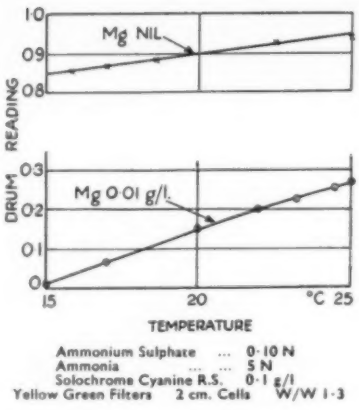


Fig. 14.—Effect of temperature on the absorption by reagent and magnesium complex.

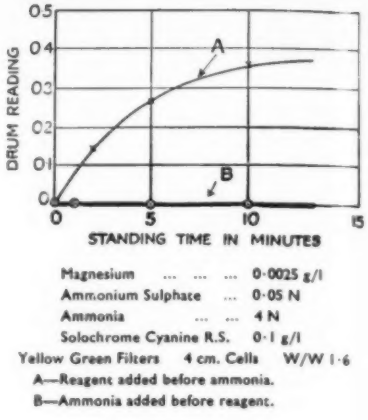

DRUM READING
A
B
STANDING TIME IN MINUTES
Magnesium ... ... 0·0025 g/l
Ammonium Sulphate ... 0·05 N
Ammonia ... ... 4 N
Solochrome Cyanine R.S. 0·1 g/l
Yellow Green Filters 4 cm. Cells W/W 1·6
A—Reagent added before ammonia.
B—Ammonia added before reagent.

Fig. 15.—Decomposition of the reagent in acid solution.

optimum *p*H can be seen to be similar to that obtained in caustic soda solutions.

Influence of the Solochrome Cyanine R.S. Concentration on the Sensitivity.—This was determined for Solochrome Cyanine R.S. concentrations of 0·05, 0·10 and 0·20 g./litre by varying the magnesium concentration, the ammonia and ammonium sulphate concentrations being constant at 5N and 0·25N respectively (*p*H 10·9). The results are given in Fig. 12 and show that, unlike Titan Yellow, the sensitivity is dependent on the reagent concentration. Thus, although increasing the ammonium sulphate decreases the sensitivity, this effect can be counterbalanced to some extent by increasing the Solochrome Cyanine R.S. concentration.

Preparation of the Reagent.—A solution of Solochrome Cyanine R.S. in water (0·05%) has a *p*H of 3·4 and it was found that this solution slowly deteriorated. A series of reagent solutions was therefore prepared adjusting the *p*H over the range 2·0 to 13·0 by additions of sulphuric acid or caustic soda. The sensitivity of the prepared reagent solution was measured under the following conditions :—

Ammonium sulphate	0·10N
Ammonia	5·0N
Solochrome Cyanine R.S. ..	0·1 g./litre
Volume	100 ml.
*p*H	11·2

immediately after preparing the reagent solution and 24 hours later.

The sensitivity is plotted against the *p*H of the Solochrome Cyanine preparation in Fig. 13 and it can be seen that the dye is unstable in neutral solutions.

Temperature Effect.—The effect of temperature on the absorption by Solochrome Cyanine R.S. and the magnesium complex is shown in Fig. 14. A rise in temperature results in a decrease in the absorption, and it can be seen that the temperature effect is more pronounced when magnesium is present. The temperatures were taken by immersing a thermometer into the solution in the cell, directly after reading on the Spekker.

Decomposition of the Reagent in Acid Solutions.—It was found that when the Solochrome Cyanine R.S. was added to magnesium solutions approximately N/10 in sulphuric acid, the colour subsequently developed decreased progressively with the time which elapsed before rendering the solution ammoniacal, indicating that the reagent is unstable in mineral acid solutions. This is shown in Fig. 15 (graph A) in which the absorption is plotted against the time interval between adding the reagent and making ammoniacal. When the ammonia was added before the reagent this phenomenon did not occur (Fig. 15 graph B).

Interferences.—Metals precipitated by ammonia will cause interference which cannot be eliminated by tartrate or citrate since traces of these inhibit the colour reaction.

Zinc was found not to interfere up to 0·10 g./litre. Small interfering absorptions were experienced in the case of nickel and copper ; the absorption being due to the formation of the green-blue ammonium complexes. The absorption by the copper and nickel are respectively about 1/50th and 1/100th that produced by the same concentration of magnesium.

It was found that in the absence of magnesium, at a concentration of 0·03 g./litre, aluminium only slowly precipitated, the speed of precipitation decreasing with the aluminium concentration. The immediate reading was not affected by 0·03 g./litre aluminium but the absorption increased as alumina precipitated.

On introducing magnesium, however, this critical concentration of aluminium rapidly decreased so that, at a concentration of 0·005 g./litre magnesium, only 0·005 g./litre aluminium was permissible if the absorption was to remain constant for 10 minutes. After this period the absorption decreased as precipitation occurred. Increasing the magnesium decreased the critical aluminium concentration still further and, at 0·01 g./litre magnesium, about 0·002 g./litre aluminium so reduced the stability period that reproducible readings could not be obtained. These tests were conducted under the conditions used for testing the reagent preparations.

The Application to Aluminium Alloys

The separation of aluminium from magnesium by caustic soda has often been observed to be incomplete. The occluded or co-precipitated alumina is dependent on the quantity of magnesia precipitated and a series of tests was conducted to determine the degree of co-precipitation.

TABLE I.—THE CO-PRECIPITATION OF MAGNESIUM AND ALUMINIUM BY CAUSTIC SODA.

Al added g.	Mg added g.	NaOH conc: N	Na Cit conc: N	Volume ml.	Al Co-precipitated g.	Mg found g.	Mg / Al ratio
0·1	nil	0·1	nil	50	0·00008	nil	—
0·1	0·002	0·1	nil	50	0·0011	0·00192	1·75
0·1	0·001	0·1	nil	50	0·00054	0·00104	1·92
0·1	0·001	0·1	nil	250	0·00057	0·00100	1·75
0·1	0·001	1·0	nil	50	0·00056	0·00100	1·78
0·1	0·001	0·1	0·2	50	0·000475	0·00084	1·76

The results are shown in Table I and the consistency of the ratio magnesium/aluminium shows the degree of co-precipitation to be remarkably constant over a wide range of conditions. In the presence of citrate, the ratio is 1·76, but the recovery of magnesium and aluminium is not complete. The results are low probably due to traces of citrate remaining with the precipitate. It is apparent from these results that the Titan Yellow reagent cannot be directly applied after a caustic soda separation.

The permissible aluminium concentration using Solochrome Cyanine R.S. decreases as the magnesium concentration increases and it is evident that, since the aluminium co-precipitated increases with the quantity of magnesia precipitated, the range of the calibration graphs that can be directly applied is strictly limited. At the higher concentrations of magnesium covered by a 1 cm. calibration graph, the aluminium remaining after a caustic soda separation interferes so strongly that the Solochrome Cyanine method is not applicable. The aluminium should be removed, therefore, by a subsequent ammonia separation, the caustic soda precipitate being redissolved in standard acid in order to control the ammonium sulphate concentration.

Direct method for magnesium in aluminium alloys (0–1.0%) (Note 1)

Method

Dissolve 0·25 g. of the sample in 20 ml. of 25% caustic soda. When solution is complete, dilute to 100–150 ml., boil, and filter. Wash with 0·01N caustic soda. Pass 50 ml. of normal sulphuric acid through the pad, wash with water, collect and dilute to 100 ml.

Sample

Transfer 10 ml. to a 100 ml. graduated flask.

Add 40 ml. water.

,, 25 ml. ammonia (S.G. 0.880).

,, 20 ml. 0·05% solution of Solochrome Cyanine R.S. (Note 2).

Dilute to 100 ml.

Blank

Transfer 10 ml. of 0·50N sulphuric acid to a 100 ml. graduated flask and continue as for the sample.

Read on the Spekker, using white light, yellow green filters, 4 cm. cells and a W/W setting of 1·6, within 15 minutes of developing the colour. Derive differences and refer to the calibration graph to obtain the magnesium content (Note 3).

Calibration

Solution A.—Balancer.

Dissolve 0·25 g. of pure aluminium in 20 ml. of 25% caustic soda and continue as described in the method.

Solution B.—Synthetic alloy (1% magnesium).

Dissolve 0·25% g. of pure aluminium in 20 ml. of 25% caustic soda, add 10 ml. of magnesium solution (Note 4) and continue as described in the method.

Prepare synthetic mixtures by varying the quantities of solutions A and B: colour, read and derive differences as described in the method and prepare a calibration graph.

Conclusion

For the successful application of the Titan Yellow method, the solution to be analysed must be free from aluminium. This is a disadvantage in the analysis of aluminium alloys, since the separation of aluminium from magnesium by caustic soda is not complete and a further ammonia separation is necessary. A method using Solochrome Cyanine R.S. has been devised, in which, over a limited range of magnesium concentration, the solution need not be completely free from aluminium. This method is readily applicable to aluminium alloys, the majority of the aluminium being separated with caustic soda.

Acknowledgment

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Note 1. The method can be easily adapted to a wider range of magnesium content by increasing the quantity of acid used to redissolve the magnesia precipitate and reducing the size of the aliquot.
e.g.—For the range 0–2·0% magnesium redissolve the precipitate in 50 ml. of 2N sulphuric acid, dilute to 200 ml. and transfer 10 ml. to a 100 ml. graduated flask.
The same calibration graph is applicable but the magnesium scale should be doubled.

Note 2. Dissolve 0·5 g. of Solochrome Cyanine R.S. in about 600 ml. of water containing 10 ml. of normal caustic soda. Dilute to 1,000 ml.

Note 3. (*a*) The temperature of the blank and the sample must be within 0·5° C.
(*b*) A deviation of ± 1° C. from the calibration temperature will introduce about ± 1% error in the magnesium content.
(*c*) By deriving differences with respect to a synthetic standard, closely comparable in magnesium content to the sample, the temperature error due to deviation from the calibration temperature, is reduced to a negligible amount.

Note 4. Dissolve 0·25 g. of pure magnesium in 100 ml. of N sulphuric acid, boil, cool and dilute to 1,000 ml.
1 ml. = 0·00025 g. magnesium.

New Factory for Oertling

Although a post-war reorganisation of their existing factory has enabled L. Oertling, Ltd., to increase their production even when compared with the war-time output, a continuing and growing demand for Oertling balances has made the provision of a larger and more modern works an urgent necessity. To meet this need, the construction of a new factory with some 33,000 sq. ft. covered area, has commenced at St. Mary Cray. It will be fully equipped with the most up-to-date machine tools for the production and finishing of balance components, and specialised instruments of recent design for the testing of balances and the adjustment of weights.

Ideal working conditions with full canteen facilities will be provided in rural surroundings and the entire factory, which is of pre-cast concrete, barrel-vault roof, north-light construction, will be temperature controlled. Air-conditioned, temperature controlled balance rooms entered through specially constructed air locks will be provided for the adjustment and standardisation of balances and weights. The Research Laboratories at present in London will be located at the factory providing increased space and facilities for the investigation of problems of balance design.

Preparation of Alumina Ware

Comments on the Methods used at R.A.E., at B.C.R.A., at Leeds University, and at N.P.L.

by Vaughan H. Stott, D.Sc.

In this article, the author comments on the series of articles, which have appeared in recent months, in which the techniques used in making sintered alumina at the Royal Aircraft Establishment, the University of Leeds, the British Ceramic Research Association, and the National Physical Laboratory have been described.

Preparation of Alumina Slip

The raw material is the same in each case, being calcined alumina as used for the production of aluminium; a typical analysis is given by R.A.E. The alkali content is believed to have a considerable influence on the behaviour of slips and on the grain growth during firing, but small ware which has been fired for half-an-hour at 1800° C. may contain less than 0·1% of alkali, calculated as Na_2O, although the value for the raw material may have been as much as 0·75%. The loss of alkali is due to volatilisation.

Ball mills are generally used for grinding alumina, but R.A.E. also had the material "micronised." Wet grinding is described by B.C.R.A. The present writer has found that steel or iron mills, when used wet, may generate hydrogen which can reach a considerable pressure. Further information would be useful.

The alumina "micronised" for R.A.E. was a little coarser than ball-milled alumina as used in N.P.L. The micronised alumina was therefore ball-milled dry for 150 hours, after which half the material was finer than 2μ. Dried casts from this finer material had a density of 2·5, as compared with 2·2 from the "micronised" alumina. A density of 2·8 has been obtained in N.P.L. with alumina such that the mean particle size (i.e. the size below which half the material is included) lay between that of the "micronised," and that of the alumina which has been ball-milled after "micronising."

The use of rather strong or hot acid ultimately results in the presence of colloidal aluminium hydroxide in the slip. A very small quantity of this substance may be beneficial, but larger quantities may seriously retard the flow of water through a partly formed casting, and may have the additional effect of reducing the density of the unfired body. The latter effect increases the possible firing shrinkage, and so may lead to warped or cracked ware. A tendency to crack may also be connected with casting and drying difficulties associated with colloidal matter, or resulting from too fine grinding.

Slip-casting

The use of hot air to facilitate and accelerate casting is mentioned only by N.P.L. It is, however, stated by B.C.R.A. that ware more than 2 mm. thick must be dried very slowly in the mould to prevent the formation of cracks; cracking is also mentioned by R.A.E. It will be realised that the production of a good slip involves compromise. A slip which is too coarse requires an extremely high firing temperature, whilst one which is too fine may give rise to the troubles mentioned above, and a slip of intermediate fineness may tend to stick to the mould. Oiling the mould may overcome the sticking difficulty, but it lengthens the time of casting. Slips yielding weak castings may be improved by a small addition of gum arabic, or other similar substance, to increase both the stability of the slip and the strength of the casting. Very thin alumina ware has been cast in N.P.L. by adding ammonium alginate to the slip. The alumina must be throughly washed before the addition of ammonium alginate is made, for otherwise the residual hydrochloric acid would react with the ammonium alginate.

The casting, from a fine slip, of very thick objects, such as balls and rods, is difficult; and it may be found that finished ware, exhibiting no external flaws, contains serious internal cracks which can be detected by using X-rays.

Preparation of Moulds

Methods of mould-making vary little, but in the past it has been important to have no appreciable external constraint on the plaster during setting, so that the pattern would not be gripped as a result of the expansion of the plaster (of the order of 0·5% linear when free from constraint). The expansion of some modern plasters is very small, and the above precaution may not always be necessary. The proportion of plaster to water can be varied considerably without producing any important effect on the slip-casting, for the porosity of a mould containing the highest practicable amount of plaster is ample. Moulds made with a relatively high proportion of plaster are less porous, but are stronger and last longer than those made with more water, but the latter are more easily made. In the present writer's experience the weight of plaster is about 1·6 times the weight of water.

Firing

No entirely satisfactory solution of the firing problem has been found, except on a small scale. The use of oxygen in a gas-furnace simplifies the attainment of high temperatures, but tends to increase the variation of temperature from point to point. Since some kind of saggar may be necessary in a small gas-furnace, a larger furnace may be needed to produce the saggar. The firing temperature for this purpose, however, need not exceed 1500° C., for by the plentiful use of fused alumina the firing shrinkage of the saggar may be kept low. Saggars with walls half an inch thick may be used many times, even when badly broken, if the pieces form a stable structure when in their proper positions. The parts may be tied together with string to facilitate placing the saggar in the furnace.

As indicated by Leeds University, alumina fired to 1700° C. for one or two hours is serviceable for many

Continued on page 218

B.I.S.R.A.'s Fifth Annual Chemists' Conference

A brief account is presented of the fifth annual Chemists' Conference arranged by the British Iron and Steel Research Association, and held at Ashorne Hill on October 3rd and 4th.

STANDARD methods of analysis of elements in iron and steel, and the recovery of sulphuric acid from the ferrous sulphate produced in "pickling" sheet steel were among the subjects discussed at the fifth annual Chemists' Conference held by the British Iron and Steel Research Association at Leamington on October 3rd and 4th, under the Chairmanship of Dr. E. Gregory, of Edgar Allen & Co., Ltd. About 130 delegates attended.

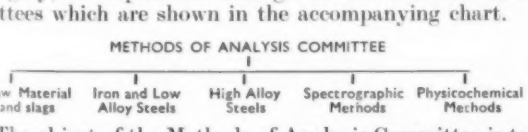
Seven papers were heard and discussed in three sessions. The first two papers were in the nature of reviews of current work :

THE SPECTROGRAPHIC ANALYSIS OF LOW ALLOY STEELS, by Mr. J. H. Oldfield (Bragg Laboratory, N.O.I.D.); THE DETERMINATION OF GASES IN IRON AND STEEL, by Mr. G. E. Speight (United Steel Companies, Ltd.).

Four papers were concerned with analytical procedures : THE DETERMINATION OF MOLYBDENUM IN LOW ALLOY STEELS, by Mr. S. W. Craven (I.C.I., Ltd., Alkali Division); THE CONFLICTING BEHAVIOUR OF MOLYBDENUM, TUNGSTEN AND VANADIUM IN ABSORPTIOMETRIC ANALYSIS, by Mr. C. H. R. Gentry (Philips Electrical, Ltd.); THE DETERMINATION OF VANADIUM IN FERRO-VANADIUM, by Mr. B. Bagshawe (Brown-Firth Research Laboratories); THE DETERMINATION OF NICKEL IN HIGHLY ALLOYED STEELS, by Mr. S. Harrison (Kayser, Ellison & Co., Ltd.).

Finally, Mr. W. Bullough (British Iron and Steel Research Association) gave a paper on THE DISPOSAL OF PICKLE LIQUOR WITH PARTICULAR REFERENCE TO CONSERVATION OF SULPHURIC ACID.

The first six of these papers were concerned with some aspects of the work for which B.I.S.R.A.'s Methods of Analysis Committee, under the Chairmanship of Dr. Gregory, is responsible through its various Sub-Committees which are shown in the accompanying chart.



The object of the Methods of Analysis Committee is to co-ordinate and centralise, as far as is economical, all research into the development and standardisation of methods of analysis. The value to the steelmaker and user alike of developing accurate and standard methods of analysis is obvious, and the extent to which the work has gone forward is shown by the fact that 18 of the parts of B.S. 1121 issued by the British Standards Institution have been worked out by this Committee or its predecessor. Such work must by its nature entail co-operation between many laboratories whose pooled experience and comparative tests are essential for the development of standard methods. In the paper on Spectrographic Analysis, for example, it is recorded that one series of tests alone involved the making and assessment of about 8,900 spectrograms systematically spread among 12 laboratories to provide completely reliable information. Many instances such as this could be taken from all the Sub-Committee's work.

This is the first time that we have been privileged to publish a report of the B.I.S.R.A. Chemists' Conference. Members of B.I.S.R.A. who attend it receive a full confidential report, and this has hitherto been considered sufficient. Now, however, the Committee's work is thought to be reaching a stage where it is of interest and importance to other industries than the iron and steel industry and its immediate associates. We are asked to state that further information is readily available to enquirers of the Committee's Secretary, Mr. J. O. Lay, at 11, Park Lane, London, W.1.

The Spectrographic Analysis of Low Alloy Steels

MR. J. H. OLDFIELD (Bragg Laboratory, N.O.I.D.) reviewed current work on "The Spectrographic Analysis of Low Alloy Steels," and a short summary of his paper follows. In showing how reproducibility had been attained to the levels shown in Table I over the ranges shown in Table II, he dealt with the three main factors in spectrographic analysis :

(*a*) the excitation of the sample ;
(*b*) the dispersion and recording of the emitted radiation ;
(*c*) the evaluation of that record.

He summarised some of the recommendations of the Methods of Analysis Committee on spectrographic analysis which are to be published shortly, and explained to the conference how they had been selected, the main consideration in each case being reproducibility.

Source Unit.—There are four possible types of source unit for exciting the spark. There is one with a high voltage, low capacity condenser with no control over the spark which may occur 2, 3 or even 4 times in one half cycle, and a similar type with a spark controlled by a mechanical device to occur only once in a half cycle.

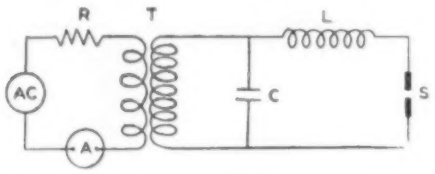
TABLE I.—SPECTROGRAPHIC ANALYSIS: STANDARD DEVIATION IN ORDER OF INCREASING ELEMENT CONTENT

Manganese		Nickel		Chromium		Silicon		Molybdenum		Vanadium		Copper	
Chem. %	S.D. %	Chem. %	S.D. %	Chem. %	S.D. %	Chem. %	S.D. %	Chem. %	S.D. %	Chem. %	S.D. %	Chem. %	S.D. %
·36	2·9	0·18	6·1	0·20	2·2	0·13	5·8	0·32	2·18	0·12	3·6	0·11	3·7
·52	1·9	0·84	1·6	0·35	2·2	0·18	3·9	0·42	1·9	0·22	2·6	0·18	5·6
·79	2·6	2·08	1·5	0·53	2·3	0·25	4·5	0·53	2·4	0·36	2·6	0·23	2·9
·21	2·1	2·92	1·9	1·72	1·4	0·39	3·1	0·95	2·4	0·46	2·4	0·31	3·2
·42	2·2	4·10	1·6	2·31	1·6	0·57	2·5	1·29	2·4	0·52	2·3	0·50	3·7
—	—	—	—	3·07	1·6	0·81	2·1	—	—	0·65	3·1	—	—

TABLE II.—SPECTROGRAPHIC ANALYSIS: ELEMENT CONCENTRATION RANGE

Element	Percentage Concentration Range
Silicon	0·05—0·80
Manganese	0·05—1·50
Nickel	0·10—5·00
Chromium	0·05—3·00
Molybdenum	0·05—1·50
Vanadium	0·03—0·65
Copper	0·05—0·50

There is the type with a low voltage, high capacity condenser, which was originally developed by the British Non-Ferrous Metals Research Association, and lastly there is the type which uses electronic means to break down the " analytical gap " between the sample and the counter electrode. There was little to choose between the various types for reproducibility after many tests, and because of its simplicity the first type mentioned, with an uncontrolled spark has been recommended. A typical circuit and some details are given as Fig. 1.



A Ammeter
A.C. Alternating Current Generator
R Adjustable Resistance
T Transformer
C Condenser
L Self-inductance
S Spark Gap

Fig. 1.—Conventional uncontrolled condensed spark.

Examination of the effects of electrode breakdown has led to the recommendation that the counter electrode should be pure graphite rod 6·5 mm. diameter, pointed to an 80° cone. The sample itself need not conform to a standard size, but should not normally be smaller than $\frac{1}{2} \times \frac{1}{2} \times \frac{1}{4}$ in. Because the method should be applicable to many shapes and sizes, no preparation other than filing to a smooth, flat surface is desirable.

Dispersing and Recording the Radiation.—As grating spectrographs are not easily obtainable in this country, detailed recommendations on the assembly and operation only of quartz prism instruments were described in the paper. It is hoped that these, together with the recommended photographic procedures, will be published with the Sub-Committee's full recommendations early in 1952.

Evaluation.—Comparison of the density of the spectral lines of the various elements with the density of appropriately selected spectral lines of iron is the method employed. For this to be effectively reproducible three things are necessary :

calibration of each plate must relate the density of any spectral line image on the photographic plate to its intensity in the radiating source ;

standardisation of each plate must relate this density value to the concentration of the element in the sample ;

density ratios of the pairs of lines selected must not change significantly over the exposure times used.

Calibration can be done in several ways ; the method recommended is chosen for its ease and simplicity. It depends on the fact that certain iron lines (listed in Table III) form a homologous series, that is they do not vary in their relative intensities whatever changes may take place in the exciting source. A primary calibration curve is obtained by means of an iron spark spectrogram made with a step sector of known angular ratio rotating directly in front of the spectrograph slit. The exposure is adjusted to give a range of density values for the line Fe 2783·696 similar to those indicated in Fig. 2. After processing by a standard procedure, readings on a non-recording densitometer (clear glass readings at full scale deflection) for each of the steps of this line are plotted against the relative intensity values calculated from the known angular ratios of the step sector and the unfiltered portion. The densities of the other lines shown in Table IV are then obtained from the unfiltered portion of the step sector spectrogram and are converted to relative intensity values by means of the curve. The line Fe 2799·3 is arbitrarily selected to have a relative intensity of 1·0.

Standardisation of each plate is carried out by first plotting a plate calibration curve. Three spectrograms of each standard steel and an iron spark spectrogram are obtained, and the densitometer scale deflections are taken for the line pairs and the iron calibration lines. The deflection values of the latter are then plotted against their relative intensity values. This plate calibration curve is then used to convert the deflection values for the line pairs into relative intensity values, and the log of these ratios is plotted against the log of the percentage concentration of the element, to produce working curves such as those shown as Fig. 3.

Experiments have shown that the density ratios of the line pairs used remain constant after the first few seconds of exposure for up to two minutes. A second factor determining the exposure time is that a suitable density range has to be obtained for the various spectral lines over the concentration percentages to be determined. The selection of the lines used for the various elements was based on their homologous nature, and on their nearness to a suitable iron line for comparison. The line pairs are given in Table IV, and the ranges of analysis attainable are summarised in Table II.

The reproducibility obtainable is given in terms of standard deviation in Table I. These results are based on a systematic series of spectrographic analyses numbering 54, of which 7 were carried out by all 12 members of the Sub-Committee concerned, and the remainder were each carried out by at least 2 members. Each member recorded at least 50 spectrograms on each sample, with not more than 5 to each plate, the member's assignment being spread over two months. Provided strict attention is paid to the details of the procedure, the high degree of reproducibility indicated in Table I should be achieved by any user of spectrographic analysis, while frequent checks of the working curves will ensure analytical accuracy.

Direct Reading.—Finally Mr. Oldfield briefly discussed direct-reading methods, where photomultiplier tubes were substituted for photographic plates. The photomultiplier consisted of a vacuum tube with a number of dynodes for amplifying the light intensity response, and the use of a diffraction grating instead of a prism avoided some of the difficulties presented by the latter in direct reading. There seemed to be a good prospect of such methods both increasing the number of elements determined and extending the ranges of concentration covered.

Discussion

During the discussion on this paper Mr. Van Someren (Murex, Ltd.), speaking as " an average

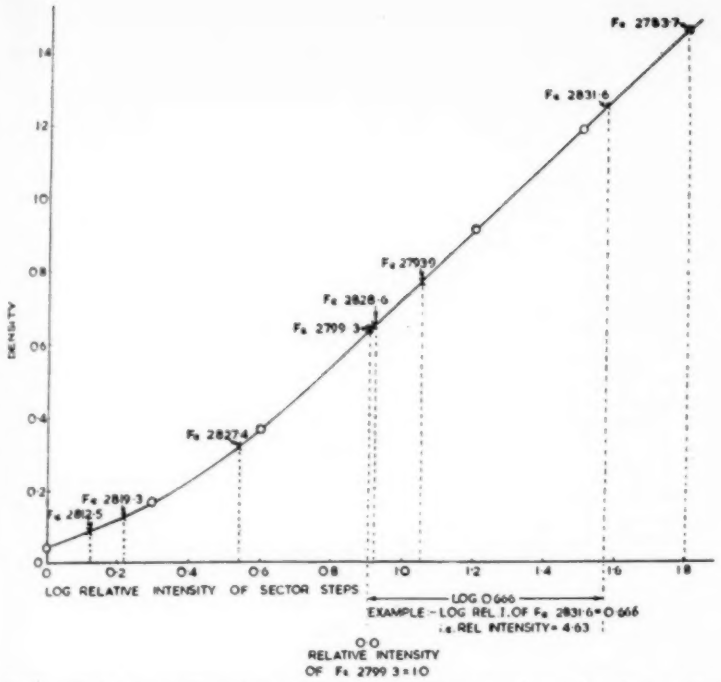


Fig. 2.—**Primary calibration curve (obtained from sector steps of Fe 2783·7 A)**

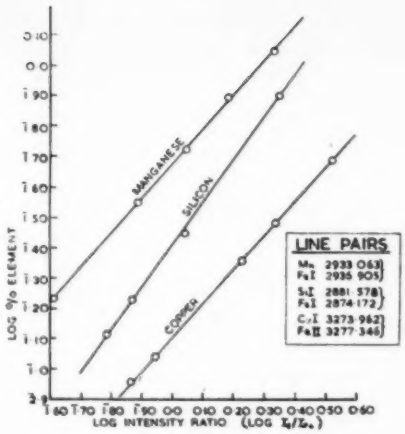


Fig. 3.—Typical working curves.

TABLE III.—HOMOLOGOUS SERIES OF IRON SPECTRAL LINES

IRON SPARK LINE
2783·7
2831·6
2793·9
2828·6
2799·3
2827·4
2819·3
2812·5

consumer," said that for various reasons it had been necessary for his laboratory to take liberties with the method outlined by Mr. Oldfield, but even so their results had confirmed the wisdom of the selection of the line pairs named. They had to use 14 gauge wire instead of the larger sample favoured by the Sub-Committee and they found it preferable to use a silver electrode in these conditions. The thinner wire tended to heat up so that the relative intensity was likely to vary more quickly than the examples given by Mr. Oldfield. They found advantage from the corona discharge given by a gramophone needle placed alongside the sample. Reproducibility was definitely improved.

Mr. Mayer (Magnesium Electron, Ltd.) raised the question of getting away from chemical standards and another speaker asked how the standard deviation of the chemical tests quoted compared with the standard deviation of two to four per cent. given for the spectrographic tests.

Mr. Oldfield replied that the corona discharge principle had been used in some of their investigations, but he was not moved to alter the Sub-Committee's recommendation. He noted that graphite electrodes gave greater reproducibility than silver electrodes though the latter did give a more intense discharge.

Mr. Oldfield thought the solution method might become useful, especially in the determination of residuals in steels. It might be advisable to get away from chemical analysis, but if it were carried out carefully, it provided sufficient accuracy. He had no standard deviation for the chemical results quoted, but they were derived from a large number of tests. Replying to other speakers who enquired if work on carbon and phosphorus determinations had been carried out, he thought very little had been done on carbon and that the combustion method in this case was adequate. Phosphorus lines, he thought, might be found free from interference if we could go further down into the ultra violet range. He thought that by direct reading with photomultiplier tubes, phosphorus and sulphur had been measured in the

TABLE IV.—SPECTRAL LINE PAIRS SELECTED

Element	Line Pair	Range
Silicon	SiI 2881·578 FeI 2874·172	0·05 to 0·60
Manganese	Mn 2933·063 FeI 2936·005	0·05 to 1·0
	MnII 3441·988 FeI 3443·878	0·50 to 2·0
Nickel	NiI 3414·765 FeI 3413·135	0·30 to 1·50
	NiI 3414·8 FeI 3417·843	0·05 to 0·30
	NiI 3101·554 FeI 3100·666	1·5 to 5·0
Chromium	CrII 3132·058 FeI 3116·663	0·05 to 0·40
	Cr 2822·412 FeI 2823·276	0·50 to 2·00
	Cr 3408 Fe 3407	2·0 to 3·0
Molybdenum	Mo 2775 Fe 2776	0·05 to 0·30
	Mo 2775 Fe 2770	0·70 to 1·5
	Mo 2816 Fe 2813	0·10 to 1·0
Vanadium	VII 3102·299 FeI 3100·666	0·05 to 0·50
Copper	CuI 3273·962 FeII 3277·346	0·05 to 0·50
	CuI 3273·962 FeII 3259·048	0·20 to 1·00

U.S.A. with the further advantage that an analysis could be done in a few minutes instead of half an hour.

A Study Group including metal physicists was now investigating discharge phenomena, one aspect being separate vaporisation of the sample and excitation of the vapour.

The Determination of Gases in Iron and Steel

Mr. G. E. Speight (United Steel Companies, Ltd.), then gave a paper on "The Determination of Gases in Iron and Steel." He pointed out that not only was it important to the manufacturer to be able to measure the amount of oxygen, nitrogen or hydrogen in his metal, but he also needed to know its form and distribution. This was often as important a factor as the quantity. The quality of steel and the physical properties of ferrous metals in general could be affected by the gas content of the solid metal, whether present in the free state, as was usually the case with hydrogen, or combined, in which condition oxygen and nitrogen are generally found.

Oxides could be found in very many combinations and permutations. In rimming and semi-killed steels it could occur as remanent FeO. Many nitrides were insoluble in ferrite and remained stable at normal rates of cooling. Others, together with free nitrogen, were soluble in ferrite and appeared in various forms in the finished steel. Their occurrence was of great importance to the sheet steel industry.

Hydrogen was not stable in steel nor was it found in chemical combination in steel. Hydrides were known to exist at high temperatures, but decomposed on cooling. A convenient method of liberating these gases was by vacuum fusion, when hydrogen, nitrogen and carbon monoxide were obtained and the quantity of each present could be measured. In this method the steel was melted at 1,600° to 1,700° C. in a carbon crucible in a vacuum. Carbon monoxide was thus formed, and at the same time the nitrides decomposed and the nitrogen and hydrogen were pumped off with the carbon monoxide and determined by standard procedures. The time taken by the whole of this process was about 1 hour, of which melting and gas extraction accounted for 12 to 20 minutes and gas analysis 20 minutes. This time could be shortened by using a mercury pump with a high backing pressure. There was a danger that any manganese and aluminium present might be volatilised in melting and their deposition in cooler parts of the apparatus would lead to absorption of carbon monoxide. This could be counteracted by a small alloying addition of tin. This method gave no information on distribution, but the "fractional" method could give some idea. This involved operating the vacuum fusion method at a range of temperatures, but did not distinguish compounds from pure oxides.

Chemical methods were necessary if full analysis of oxygen distribution was required. In one group of these methods the iron is dissolved away, leaving the non-metallic inclusions. The solvents used were alcoholic iodine or chlorine, but this was a slow method, which necessitated treatment at an elevated temperature for up to 12 hours. A more usual chemical method was heating the specimen to 1,150/1,200° C. between sheets of aluminium for about 1 hour. Alumina was thus formed in the steel by diffusion and could be determined. This was more effective for killed steels than for rimming or semi-killed. Oxygen could also be determined by evaluation of oxide inclusions, by adding aluminium to the liquid metal and separating the alumina present after solidification, or assessed by characteristic indications during furnace operation.

Discussing the "bomb" method of oxygen evaluation, Mr. Speight said that recently there had been doubts as to its accuracy. In this method a receptacle in two halves clamped together, containing an amount of aluminium foil, and closed at the top by a sheet steel cover, was lowered into the steel bath. When the cover was melted steel entered the receptacle, was killed by the aluminium and solidified ready for use as a test sample. It was thought that when the carbon content of the bath was high the carbon/oxygen reaction might be modified by the cooling effect of the bomb and give variable results. He thought this method should not be used where carbon contents were likely to be in excess of 0·2%.

Another practical problem that arose in the melting shop was the danger of losing oxygen with carbon during the few seconds of sampling. It was necessary to kill the sample with aluminium. The alumina must be measured and the analysis "worked backwards" from that measurement.

The evaluation of hydrogen by vacuum fusion was satisfactory. Alternatively as hydrogen evolved very quickly at 650° to 750° C., solid samples could be used to measure the total content by heating to that temperature.

Chemical methods for the evaluation of nitrogen had been well established for 100 years, and had recently been improved by the introduction of steam distillation. It appeared important to distinguish the mode in which nitrogen appeared, and to separate the insoluble nitrides from the soluble fraction, carrying out separate analyses for these.

Mr. Speight concluded by emphasising the importance of attention to sampling techniques, to ensure reliable interpretation of the analysis figures obtained.

Discussion

In the discussion on Mr. Speight's paper Mr. T. Dennison of the British Iron and Steel Research Association suggested that developments during the next 10 years would include methods of determining the modes of oxygen occurrence. B.I.S.R.A.'s Chemistry Department, for example, was working on the solubility of oxygen in iron, which was nil if the iron was pure. This solubility in fact depended on the purity of the iron, or the presence of alloying elements.

He asked how Mr. Speight distinguished combined nitrogen from uncombined. Mr. Speight replied that he used 1 : 4 sulphuric acid, separated the insoluble residue in a centrifuge, and analysed the clear solution. The residue could then be treated with fuming sulphuric acid and analysed. The nitrides most usually found were those of zirconium, titanium, boron and niobium.

Other points raised included the necessity of using high vacuum and high temperature for vacuum fusion if titanium were present in appreciable quantities, and the extent to which oxygen figures from spoon samples might read low. Mr. Speight pointed out that the bomb method had been developed to enable the sample to be taken without atmospheric contact, but he thought fears of inaccuracy from spoon samples were exaggerated, especially if a double dip was taken, the first dip heating the spoon and covering it with slag.

The Determination of Vanadium in Ferro-Vanadium

MR. B. BAGSHAWE (Brown-Firth Research Laboratories) gave a paper on "The Determination of Vanadium in Ferro-Vanadium." This was one of a series of three investigations on the determination of vanadium in (*a*) carbon and low alloy steels and irons, (*b*) in highly alloyed steels, and (*c*) in ferro-vanadium alloys. The method which he described for the last named purpose was recommended after 4 methods and 7 or 8 types of procedure had been examined. It consisted, in principle, of dissolving the sample in sulphuric and nitric acids and oxidising the vanadium to the quinquivalent condition with potassium permanganate in cold solution. Excess of potassium permanganate was reduced with sodium nitrite and sulphamic acid was used to destroy the excess of nitrite. The oxidised vanadium was titrated with ferrous ammonium sulphate and potassium dichromate, using barium diphenylamene sulphonate as indicator. The method was suitable for all grades of ferro-vanadium alloy other than high carbon or very high silicon alloys. High carbon/silicon alloys were rarely used in British practice but if such alloys were encountered the procedure could still be applied although a modified method of decomposition would be necessary. Tests had shown reproducibility to be $\pm$ 0·25% vanadium.

The Determination of Nickel in Highly Alloyed Steels

MR. S. HARRISON (Kayser Ellison & Co., Ltd.), gave a paper on "The Determination of Nickel in Highly Alloyed Steels." This work was directed to extending B.S.1121 : Part 6 : 1948 (Nickel below 0·5% in carbon and low alloy steels) and B.S.1121 : Part 2 : (Nickel in permanent magnet alloys). The investigations had particular reference to the interference of copper and cobalt.

Work on the use of the sodium salt of dimethylglyoxime had made possible the formulation of the conditions required for complete precipitation of nickel in the presence of copper. These were an excess of reagent (a triple amount) and a closely controlled temperature of 90° C.

Work on precipitation of nickel in the presence of cobalt indicated that a method might be evolved involving precipitation at 90° C. and an excess of hydrogen peroxide might be effective if the cobalt content did not exceed 0·15 g.

The Determination of Molybdenum in Low Alloy Steels

MR. S. W. CRAVEN (I.C.I. Ltd., Alkali Division), gave a paper on "The Determination of Molybdenum in Low Alloy Steels." After examination of various methods of determination, alpha-benzoinoxime precipitation had been selected as the most reliable means of separating molybdenum from a steel solution. Traces of molybdenum might be lost in tungstic acid residues, however, and tungsten passing into solution was co-precipitated with the molybdenum complex.

The principle of the method evolved to overcome this limitation was that the molybdenum was precipitated from a hydrochloric acid solution of the sample with alpha-benzoinoxime. The precipitate was re-dissolved and molybdenum, after conversion to thiomolybdate, was separated as sulphide from a solution in which the precipitation of tungsten was prevented by the presence of tartrate ions. The sulphide precipitate was ignited and weighed as molybdic oxide.

This method was effective over the molybdenum range 0·02 to 2·0%. It was a slow method, but gave good reproducibility on samples containing up to 0·5% of tungsten.

The Conflicting Behaviour of Molybdenum, Tungsten and Vanadium in Absorptiometric Analysis

MR. C. H. R. GENTRY (Philips Electrical, Ltd.), gave a paper on "The Conflicting Behaviour of Molybdenum, Tungsten and Vanadium in Absorptiometric Analysis." Both molybdenum and tungsten could be determined photometrically as the thiocyanates of lower valency states of the elements. For the determination of tungsten it was essential that some complexing agent, such as phosphoric acid, should be added to keep the tungsten in acid solution ; but the presence of phosphoric acid was not necessary in the molybdenum determination. Tungsten required a stronger reducing agent to give the required lower valency, and thus, although a stannous salt in a sulphuric acid solution was suitable for the molybdenum determination, for tungsten it was essential to carry out the reduction with stannous chloride in strong hydrochloric acid medium.

The amber coloured thiocyanate of 5-valent molybdenum tended to fade, but this could be prevented by the addition of, for example, perchloric acid. Under certain conditions, iron interfered in the determination of molybdenum but the interference could be prevented by the addition of titanic sulphate. Vanadium and tungsten might also interfere, the former giving a blue thiocyanate of the 4-valent element, the effect of which was slight, but necessitated a small correction when the vanadium content was appreciable. As objections had been raised to the use of phosphoric acid to keep tungsten in solution, a preliminary separation of tungstic acid was preferred by the Physic-Chemical Methods of Analysis sub-Committee in their latest molybdenum method.

For the determination of tungsten, a mixture of titanous chloride and stannous chloride as the reducing agent had proved very satisfactory. The measurement of the absorption of the yellow tungsten thiocyanate solution was preferably made at 435 mμ where molybdenum, which formed a thiocyanate complex of the 3-valent element, did not interfere. Vanadium was reduced in this method to the 2-valent state which had an intensely yellow thiocyanate, interfering seriously with the determination of the tungsten.

The Disposal of Pickle Liquor

The last paper discussed by the conference was on "The Disposal of Pickle Liquor, with Particular Reference to Conservation of Sulphuric Acid," by MR. W. BULLOUGH (British Iron and Steel Research Association).

Mr. Bullough first discussed acid economy in the batch pickling process. He showed that from a knowledge of (1) the amount of acid fed into the pickling machine, (2) the volume and composition of the waste liquors, and (3) the average composition of the second pickle, it was possible to calculate what proportion of the acid was (1) converted to ferrous sulphate, (2) lost in waste liquors and (3) lost by the plates dragging out pickle liquor from the second tank, and transferring it to the wash tank.

From the analysis results from three pickling machines it was shown that on the average, 5·17 lb. B.O.V. (78% sulphuric acid were consumed per basis box of plate. Various means could be employed to reduce this figure. These comprised (*a*) adequate inhibition to reduce acid attack on base metal, (*b*) working down pickle liquor to give a waste liquor low in acid and high in iron content (though the iron content of the second pickling tank should be kept low enough to avoid hydrolysis of the ferrous sulphate, and subsequent staining of the plates), (*c*) increasing the time the plates were allowed to drain over the tanks, and even lightly spraying them with water and allowing them to drain again. To reduce drag-out still further, the rinse water could be used to spray the plates when taken out of the second pickling tank. Mr. Bullough quoted a case where observance of these precautions had reduced acid consumption from 5·17 lb. to 4·7 lb. B.O.V. per basis box, a saving of 11%. To give effect to these recommendations continuous recovery of liquor from tank 1 was suggested to give a pickle liquor of constant acidity and iron content. Rinse water should enter tank 2 and displace an equal volume of tank 2 liquor into tank 1—the volume of rinse water being such that the recovery system would not overflow. The acid and iron contents of tank 2 would gradually rise to constant values; the values being dependent on the volumes of rinse water and drag-out and on the composition of tank 1 liquor. Mr. Bullough showed how these values might be calculated.

Continuous strip was usually pickled in one line of four or five tanks with the pickle liquor cascading from tank to tank, and with the strip running counter-current to the pickle. It was essential to provide a highly active pickle, and in pickling lines where recovery is not practised, it was usual to add acid of about 20% w/w strength at the outgoing strip end and discharge the pickle at about 5% w/w acid; 5% w/w iron content at the ingoing strip end. Drag-out acid could be minimised by fairly straightforward means, and the largest loss of acid was in the waste liquor. Recovery was necessary to effect much acid economy.

Where ferrous sulphate was crystallised from the spent liquor and the mother liquor was returned to the pickle tanks it was not necessary to work the pickle down to a low acidity in order to save acid so long as drag-out was kept low. Ferrous sulphate could be crystallised as the heptahydrate by cooling the liquor or as the mono-hydrate by evaporating the liquor to high acid strength. Heptahydrate could be subsequently dried to monohydrate and if it was required to convert the ferrous sulphate to sulphuric acid it was necessary to have the crystals in the latter form. It could then be converted to sulphuric acid in pyrites burning acid plants, or in chamber or contact acid plants, though all these methods had to be worked on a large scale to be economic.

B.I.S.R.A., however, had developed an autoxidation method of producing dilute acid (possibly up to 40%). In this process ferrous sulphate was roasted with coke to give iron oxide and sulphur dioxide, which was absorbed in water and catalytically oxidised to sulphuric acid. Mr. Bullough gave a description of a pilot plant to operate this process in a works in South Wales, at the rate of 1 ton of dilute acid per day. If the process were a success, full scale plants would probably operate at 10 or 20 times that rate.

Sunvic Factory at Harlow New Town

CONSEQUENT upon the ever-increasing demands for their products, Sunvic Controls, Ltd., have acquired a new factory on the industrial estate at Harlow New Town. The works and offices previously located at 132/135, Long Acre have removed to Harlow, where production has already commenced. The Sales Department is now situated at 132/135, Long Acre, London, W.C.2, and enquiries and orders should in future be sent to that address. The Registered Office remains at Sunvic House, 10, Essex Street, Strand, London, W.C.2.

Preparation of Alumina Ware

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purposes, and warping is reduced by not firing too hard. It may, nevertheless, be sometimes desirable to fire at temperatures as high as 1800° C. or more, and this is very expensive for all but small articles. The effect of increasing the period of maximum temperature is more marked with alumina than with argillaceous material, and a long treatment at 1750° C. seems to produce less warping, for a given final density, than a shorter treatment at a higher temperature. Ryschkewitsch has stated that, for a given final density, the higher firing temperatures yield the finer crystal structures. The present writer's experience suggests that crystal growth is too erratic for this conclusion to be regarded as established.

Acknowledgment

The above comments have been prepared as part of the research programme of the National Physical Laboratory and are published by permission of the Director of the Laboratory.

Change of Address

W. & J. GEORGE & BECKER, LTD., announce that the address of the London Area and Export Sales Offices and Warehouse is now Nivoc House, Ealing Road, Alperton, Wembley, Middlesex. (Telephone: Perivale 3344; Telegrams and Cables: Nivoc, Wembley).